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OFFICIAL PUBLICATION OF THE PROCEEDINGS, AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS

American Dyestuff Reporter was founded in 1917 and the Textile Colorist and Converter (formerly Textile Colorist) was incorporated with it in January, 1949. Published every other Monday, by the **HOWES PUBLISHING COMPANY, INC.**, 44 East 23rd Street, New York 10, N. Y. **C. R. Howes**, President; **Ernest J. Finan**, Executive Vice-President; **Norman A. Johnson**, Vice-President & Treasurer; **Myron D. Reeser**, Vice-President; **Clara Patrick**, Secretary.

Telephone SPring 7-9364

AMERICAN
DYESTUFF REPORTER
DEVOTED TO TEXTILE WET-PROCESSING

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AMERICAN
DYESTUFF REPORTER

Volume 48 | September 7, 1959 | Number 18

SOME FACTORS INVOLVED IN "WASH-AND-WEAR" WOOL

JOHN F KRASNY, HAZEL E HARRIS, GEORGE H LOURIGAN*, and ELEANOR D WHITE

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The "wash-and-wear" characteristics of wool fabrics were evaluated on the basis of three factors: shrinkage, mussiness, and fuzz formation in laundering. The methods used to determine these factors are described and critically evaluated. It was found that several of a group of randomly selected commercial wool fabrics were, by reason of their construction, reasonably dimensionally stable in machine laundering followed by either air or tumble drying. Those fabrics which showed some shrinkage in air drying shrank considerably more during tumble drying. Some commercial wool fabrics were perfectly smooth after machine laundering and tumble drying, but not after air drying; most wool fabrics studied would have satisfactory ratings using "wash-and-wear" standards presently used for cellulosic fabrics. In some fabrics, a considerable increase in surface fuzziness was observed after laundering. Chlorination reduced both shrinkage and formation of fuzz in laundering and tumble drying.

INTRODUCTION

HARRIS Research Laboratories have undertaken a research project sponsored by the Wool Bureau, and the International Wool Secretariat to determine the effects of fiber and yarn properties and fabric construction on the felting shrinkage of wool materials, especially during home laundering with modern washing machines. This work has shown that felting shrinkage of untreated materials is much less serious in modern washers than in pre-war types because of such factors as slow agitation and spinning speeds, and short washing cycles (*1*). Studies of various fabric constructions also showed that felting shrinkage is related to fabric compactness (*2*). From these results it became apparent that, by taking advantage of construction features and/or chemical processes available for elimination of wool shrinkage (*3*), felting shrinkage as such was a much less serious problem today than in the past. Thus, consideration of wool for "wash-and-wear" end use became technically feasible, and this paper attempts to analyze some of the problems facing wool as a "wash-and-wear" fiber.

The growth of the "wash-and-wear" or "easy-care" garment market has been indeed noteworthy. It is estimated that over two billion yards of cotton goods were treated in 1958 (*4*). Wool materials have not been a factor in this market because, traditionally, wool garments are dry-cleaned. However, with the availability of washable wool, there is no reason why it could not play a role in the "wash-and-wear" field. Certainly, shrink-resistant wool socks and sweaters are, broadly speaking, "wash-and-wear" items. It is clear that each fiber presents unique problems when it is used in "wash-and-wear" fabrics, and wool is no exception. For example, cotton fabrics muss easily in laundering and drying, while this problem is much less serious with wool, as will be discussed below. On the other hand, cotton does not fuzz significantly during laundering, whereas many wool fabrics fuzz considerably, and synthetics, when used in fabrics of wool-like character, frequently fuzz or pill.

The term "wash-and-wear", as it is commonly used, refers to the mussiness or smoothness of a garment after laundering and drying. The present paper discusses also shrinkage and fuzziness, properties which are as important as mussiness in considering washable wool fabrics. The effects of variables in laundering and tumble drying on all three of these "wash-and-wear" characteristics are discussed, and typical results are presented in this paper.

* Union Carbide Chemicals Co

METHODS

RELAXATION OF FABRICS——All fabrics were relaxed by immersion in a 0.1 percent solution of nonionic wetting agent at room temperature for one hour and air dried in the absence of tension.

SHRINKAGE MEASUREMENTS——Marks 10 inches apart were made on the relaxed fabrics. Shrinkage measurements were made by means of a measuring template, which had single lines etched on its lower face on two sides of the square, and sets of 30 lines, 1/10 of an inch apart, on the two opposite sides. These lines were marked with the corresponding percent shrinkage value. The single base line was aligned with one set of marks on the fabric, and the shrinkage determined by finding the line which aligned with the opposite marks.

The correlation between results obtained by this rapid method and by averaging the distances measured between three pairs of points in the warp and filling direction with a ruler was good; the difference in results obtained by the two methods in 22 fabrics of widely varying shrinkage did not exceed one percent.

The area shrinkage (A), where

$$A = \text{warp shrinkage} + \text{filling shrinkage} - \frac{\text{warp shrinkage} \times \text{filling shrinkage}}{100} \quad \text{(all in \%)}$$

is shown in all tables of this paper.

MUSSINESS RATINGS——For

TABLE I
Construction of wool fabrics

Fabric No.	Type	Weight (oz/sq yd)	Weave	Yarns per inch		Yarn no. worsted count		Yarn twist, tpi			
								Single		Ply	
				W	F	W	F	W	F	W	F
30	Flannel	6.9	2/2 Twill	55	44	2/42	1/12	12Z	12Z	15S	—
49	Dress goods	6.1	Plain	34	37	1/14	1/13	11S	12S	—	—
								12Z	13Z		
55	Tropical worsted	7.3	Plain	53	50	2/32	2/30	13Z	12Z	13S	13S
56	Gabardine	8.1	2/2 Twill	123	73	2/54	2/56	12Z	16Z	19S	16S
57	Tropical worsted	6.9	Plain	56	44	2/36	2/30	9Z	13Z	16S	12S
59	Gabardine	7.3	3/2 Twill	127	96	1/33	1/40	19S	18Z	—	—
60	Worsted	6.1	2/2 Twill	72	68	1/24	1/20	11Z	13Z	—	—
63	Unfinished wool	7.6	2/1 Twill	35	27	1/9	1/9	7S	8S	—	—
65	Serge	11.7	2/2 Twill	60	60	2/24	2/22	9Z	10Z	9S	9S
74	Flannel shirting	5.9	Plain	40	36	1/16	1/15	15S	12S	—	—
77	Flannel	8.3	2/1 Twill	44	48	1/14	1/14	10S	13S	—	—
81	Flannel shirting	8.4	2/2 Twill	44	48	1/14	1/13	10S	8S	—	—
82	Gabardine	7.0	2/1 Twill	73	53	1/20	1/21	16S	14S	—	—
83	Gabardine	7.6	3/2 Twill	98	61	1/24	1/23	11S	12S	—	—
84	Tropical worsted	5.7	Plain	59	47	1/21	1/18	14S	—	—	—
								11Z	13Z	—	—
86	Flannel	6.4	2/1 Twill	60	59	1/20	1/21	16S	13S	—	—
113	Flannel	7.2	2/1 Twill	66	60	1/22	1/19	14Z	15Z	—	—
129	Gabardine	7.2	3/2 Twill	116	79	1/29	1/29	18Z	18S	—	—
131	Flannel	6.9	2/1 Twill	55	55	1/18	1/18	12Z	16S	—	—

rapid rating of large numbers of fabrics for mussiness or "wash-and-wear" appearance, as the word is now generally used, the samples were placed on a long table, 30-inches high, with a light source extending behind the samples for the whole length of the table and shielded from the eyes of the observers. The angle of incidence of the light on the fabric surface was approximately 20 degrees. The observers viewed the fabrics standing up, usually about three to four feet from the samples. Three observers rated each sample about one hour after tumble drying. The ratings used here included creases as well as relatively small deviations from smoothness, such as cockled appearance.

The best rating, 5, of the common "wash-and-wear" scales (5) does not indicate a perfectly smooth fabric but various moderate or low degrees of mussiness. It was thus necessary to introduce a rating of 6 for smooth wool fabrics. In fact, the difference between some of the very smooth laundered and tumble dried wool fabrics found in this work and the present commonly used 5 rating may be more than one step, but it was felt that the extension of the scale to 6 would serve the purpose of the present paper. A rating of 5 on the scale used here is "very slightly mussy"; 4, "slightly mussy"; 3, "mussy"; and 2 represents the mussiest wool fabrics found, which appeared, however, still considerably better than cellulosic fabrics commonly rated 2 or 1. Thus, the present scale is not directly comparable to the usual "wash-and-wear" ratings, but it shares with them the concept of high numbers representing smooth fabrics.

Initially, the photographs of the "wash-and-wear" fabric scale by Rawlings (6) were used in rating the mussiness. These, however, were abandoned because of the difficulty in comparing the wool fabrics, which varied widely in texture and color, with photographs of synthetic fabrics. It was found that ratings of 12 fabrics by six observers obtained with standards agreed closely with ratings obtained without standards, but rating without standards was faster and easier for most observers.

FUZZINESS RATING——Laundering and especially tumble drying of wool fabrics often causes a raising of fibers, or fuzz formation, which is considered objectionable. This was evaluated by two methods: by compressibility measurements and by visual comparison with standards.

The Schiefer Compressometer (7) was used to determine the fabric thickness at 0.01 pounds per square inch and at regularly increasing pressures until the flattening point, ie, the point at which 0.5 pounds per square inch increase in pressure decreased the thickness by less than five mils, was reached. The difference in thickness at 0.01 pounds per square inch and at the flattening point pressure was noted for the relaxed and for the laundered fabric, and assumed to be a function of the fuzz of the fabric. This difference can then be taken as a measure of the increase in fuzziness due to laundering.

The visual evaluation of fuzziness was performed by draping the fabrics over the edge of a long vertical plywood sheet, 1/4-inch thick, so that the warp and filling were at an angle of approximately 45 degrees to the edge of the sheet. A light source illuminated the fuzz at the fabric fold from behind. Standard fabrics were used in this comparison, ranking from 1—very fuzzy (a fuzzy blanket) to 6—no fuzz. This method of rating was

TABLE II
Effect of laundering conditions on "wash-and-wear" characteristics of wool fabrics

Laundering conditions	Mild	Severe
Load	3 lbs	8 lbs
Water level	Normal	Normal
Agitator and spinning speed	Slow[1]	Fast[1]
Laundering time (soaping cycle)	3 min	10 min
Water temperature	Approx 100° F	Approx 130° F
Washing machine	Agitator type, Maytag 1957, Model 140	
Dryer	Electric, drum, Maytag 1957, Model 640W	

"Wash-and-wear" characteristics after five launderings and air drying cycles

Fabric No.	Area felting shrinkage		Mussiness rating		Fuzz formation in laundering: Visual rating[2]		Fuzz formation in laundering: Increase in thickness	
	Mild (%)	Severe (%)	Mild	Severe	Mild	Severe	Mild (mils)	Severe (mils)
55	1	4	4.4	2.6	0.2	1.0	2	25
129	5	14	3.2	2.3	0.4	1.3	18	46
131	9	30	3.3	2.3	1.6	1.6	49	71
113	12	30	2.9	2.2	0.6	0.8	32	50

[1] Slow: "Modern Fabrics" setting of washing machine; Fast: "Regular Fabrics" setting (4).
[2] A low value indicates little change due to laundering.

TABLE III

Effect of tumble drying variables on shrinkage, mussiness and fuzziness of wool fabrics

Five laundering and tumble drying cycles

Temperature[1]	Duration[2]	Load (lbs)	Removed from Dryer	Fabric no.	65	60	129	131	113	Average
Low	long	3	immediately	Area felting shrinkage (%)	2	3	9	16	28	11.6
				Mussiness rating	5.8	3.6	4.1	5.0	3.2	4.3
				Fuzz formation in laundering[3]	0.7	1.4	0.7	1.1	−0.3	0.7
High	short	3	immediately	Area felting shrinkage (%)	3	4	8	16	22	10.6
				Mussiness rating	5.8	3.0	3.6	5.3	3.3	4.2
				Fuzz formation in laundering[3]	0.6	1.5	0.5	1.0	0.3	0.8
High	short	3	after one hr	Area felting shrinkage (%)	3	3	7	14	20	9.4
				Mussiness rating	5.3	3.0	3.3	3.7	3.3	3.7
				Fuzz formation in laundering[3]	0.4	1.0	0.2	0.7	0.2	0.5
Low	long	8	immediately	Area felting shrinkage (%)	4	6	15	26	41	18.4
				Mussiness rating	4.6	3.1	3.6	4.4	2.8	3.7
				Fuzz formation in laundering[3]	0.8	1.4	0.7	1.0	−0.1	0.8
High	short	8	immediately	Area felting shrinkage (%)	4	4	12	23	30	14.6
				Mussiness rating	5.4	3.0	3.3	4.8	2.8	3.9
				Fuzz formation in laundering[3]	0.9	1.4	0.7	1.1	0.1	0.8

[1] Temperature: "Low"—approximately 140° F, "High"—approximately 172° F.
[2] Duration: 3-lbs load: "Short"—35 minutes and 5 minutes cooling period.
"Long"—60 " " " " "
8-lbs load: "Short"—80 " " " " "
"Long"—130 " " " " "
[3] A low value indicates little change due to laundering.

adopted to conform with the "wash-and-wear" or mussiness ratings in which a high number means a good fabric. Fabrics could be arranged in order of increasing fuzziness or placed next to the appropriate standards for rating. The visual method permits evaluation of the evenness of the fuzz and differentiation between relatively few, long fibers or many short fibers emerging from the fabrics. It is compared with the thickness increase method under "Results and Discussion" below under the subtitle "Comparison of Subjective Fuzziness Ratings with Compressometer Measurements".

CHLORINATION TREATMENTS———The wool fabrics were chlorinated by the Stevenson-Wolsey process (*8*), which had been modified to permit the use of laboratory equipment. The treating solutions contained one percent nonionic wetting agent (owf), 10% $CaCl_2$, and various amounts (one, two, three and four percent) of $KNnO_4$ and NaOCl. The permanganate and hypochlorite were added in three equal portions at ten-minute intervals after the fabrics had been wet-out in the wetting agent and calcium chloride. During the treatment, the fabrics were circulated as a continuous belt through the treating liquor and the rolls of a laboratory padder. When at least 90% of the available chlorine had been exhausted, ie, 50 minutes after the last addition of hypochlorite and permanganate, the fabrics were rinsed and subjected to an antichlor treatment in sodium bisulfite and acetic acid. The treated samples were then given a two-hour running rinse, centrifuged, dried, and relaxed in the usual manner.

MATERIALS

The fabrics used in this work represented a wide range of commercially available untreated wool fabrics. Their construction, after relaxation, is shown in Table I.

RESULTS AND DISCUSSION

EFFECT OF LAUNDERING CONDITIONS———The effect of various laundering conditions obtainable with a modern home washing machine on the shrinkage of wool fabrics was described in detail in a previous paper, and a satisfactory, mild laundering procedure was recommended (*1*). Table II gives the details of this mild and of a severe laundering procedure, and compares their effects not only on shrinkage but also on mussiness and fuzz formation of several wool fabrics. As expected, the mild laundering conditions were preferable from the point of view of optimizing these properties, and they were used in all the work described below.

The modern tumble dryer, like the modern washing machine, permits a rather wide choice of conditions. Experiments in which these conditions were varied systematically led to the results shown in Table III. In general, these variations did not lead to large differences in results, indicating the relative insensitivity of wool fabrics to drying conditions. The optimum results were obtained when the load was low (three pounds) and a combination of high temperature and short drying time was used. The mussiness of three of the five wool fabrics was not affected by leaving the fabrics in the tumble dryer for one hour after it had stopped, but two appeared mussier when thus treated. This is in contrast to instructions for laundering of fully synthetic fabrics, which generally recommend removal of the garments from the dryer immediately after it stops (*9*).

COMPARISON OF EFFECT OF AIR AND TUMBLE DRYING———Figure 1 compares the results obtained when swatches of a representative range of commercially available wool fabrics, none of which was chemically treated for shrink resistance, were laundered and air dried and when swatches of the same fabrics were laundered and tumble dried. Three findings are of interest.

1) Fabrics which by reason of their construction alone were dimensionally stable in laundering and air drying were also dimensionally stable when tumble dried. However, in the case of those fabrics which showed area felting shrinkages of over five percent in 10 laundering and air-drying cycles, tumble drying caused substantial additional shrinkage.

2) Many fabrics were surprisingly smooth, especially after tumble drying. In fact, a mussiness rating of 6 was used in this work because some wool fabrics were, when tumble dried, at least one step smoother than fabrics given the best rating, 5, by presently used "wash-and-wear" standards (*5*).* The reduction in mussiness due to tumble drying varied greatly from fabric to fabric, the most dramatic improvements being illustrated in Figure 2. It is also of interest to note that Fabrics 55, 84, 65 and 82, which exhibited rather low shrinkage, also were low in mussiness and would be satisfactory for wear with

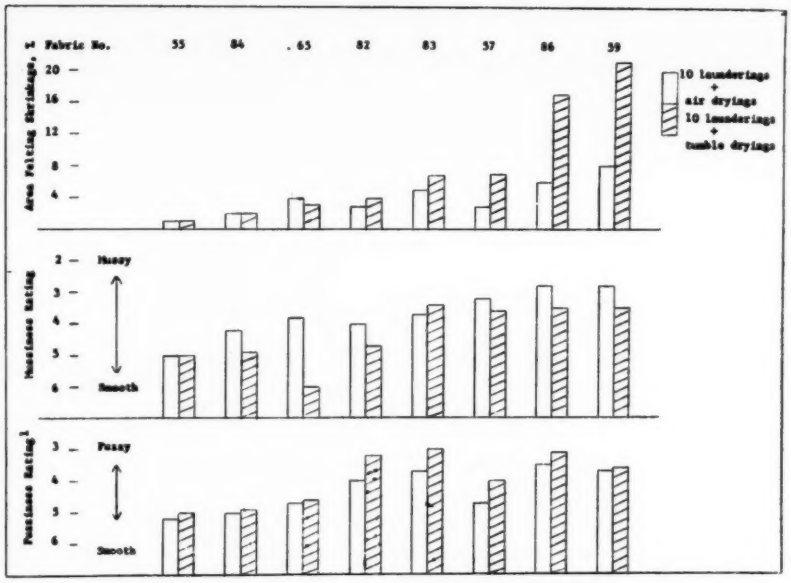
* The mussiness and fuzziness ratings are plotted with 6 (best) on the bottom, 2 (worst) on the top in Figure 1, so that a short bar represents a good result, like in shrinkage.

little or no ironing after tumble drying.

3) The fuzziness was generally worse after tumble drying than after air drying. Again, the above-mentioned fabrics with low shrinkage and low mussiness were also less subject to fuzz formation. Thus there were several among these commercial, untreated wool fabrics (chosen to represent a wide range of shrinkage) which approached "wash-and-wear" status rather closely.

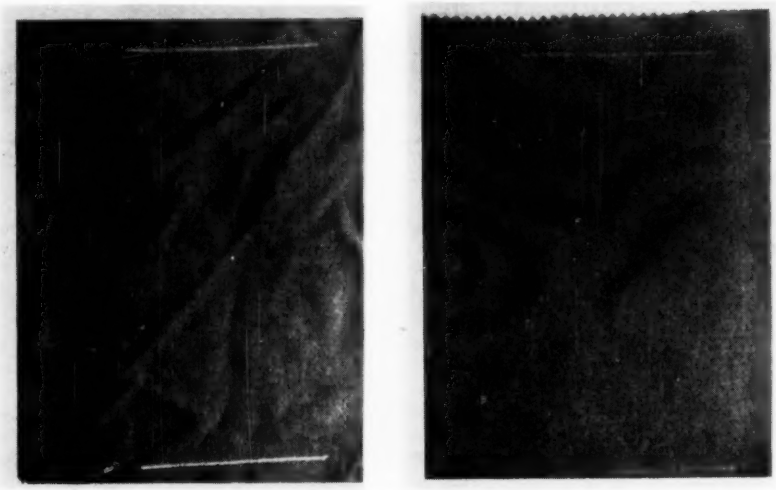
COMPARISON OF RESULTS OBTAINED ON SWATCHES AND ON GARMENTS; RESULTS OF LIMITED SERVICE TESTS —— One question which arises in evaluating laundering experiments concerns the correlation between data obtained on swatches with those observed on garments. Table IV compares results obtained on swatches 12 by 12 inches in size and garments made from the same fabrics. In general, the garments shrank to about the same extent as, or slightly more than, the swatches. The mussiness of the garments and swatches was about equal except in the case of the untreated shirting fabric after tumble drying; in this case, the shirt was so puckered at the seams and distorted, perhaps because of unbalanced warp and filling shrinkage, that it appeared extremely mussy as compared to the swatches. The mussiness rating of Fabric 83, garment and swatch alike, was affected by the pucker caused by inordinate shrinkage of individual filling yarns. In this fabric, considerably less fuzz was raised on the swatch than the garment after laundering; in all other cases the difference was small.

Limited service tests with wool skirts and shirts produced some interesting qualitative results. The fuzz created in laundering was not significantly reduced by ironing and wearing of the garments between laundering cycles as compared to swatches which were laundered only. Double layers of fabrics, such as hems or seams, require specific attention in evaluating wool fabrics for "wash-and-wear" characteristics; in these places, excessive shrinkage, fuzziness and/or abraded areas and puckering sometimes appeared before the garment became unserviceable due to tight fit. All these deficiencies were much less apparent in a dimensionally stable fabric than in one with even moderate shrinkage (approximately seven percent area shrinkage in 10 laundering cycles). In general, it appeared that, for "wash-and-wear" wool fabrics, dimensional stability and perhaps fuzziness control will have to be achieved by choice of proper combination of structure and /or treatment, while mussiness apparently presents a much smaller problem in many types of wool fabrics than in, eg, cellulosic fabrics.



[1]Absolute fuzziness rating of laundered fabrics. A long bar indicates high fuzziness.

Figure 1
Effect of air and tumble drying on "wash and wear" characteristics of wool fabrics



Fabric No. 65 after 10 launderings and air drying cycles. Mussiness rating: 3-8

Fabric No. 65 after 10 launderings and tumble drying cycles. Mussiness rating: 6.0

Figure 2
Effect of air and of tumble drying on the mussiness of wool fabrics

EFFECT OF NUMBER OF LAUNDERING CYCLES —— Another question which often arises in conjunction with laundering tests concerns the number of launderings necessary to obtain a valid evaluation of fabrics. The present work was not designed to evaluate the stability of dyes or chemical finishes, but the minimum number of launderings required for reliable comparison of shrinkage, mussiness and fuzziness of various wool fabrics was of interest. Figure 3 shows the area shrinkage of eleven commercial fabrics during 100 launderings and air-drying cycles. Only in two cases was there a crossing over of the lines after the 10-cycle mark, and the shrinkage after 100 cycles was generally two to three times as great as that at 10 cycles. The fabrics, unlike cotton fabrics, did

TABLE IV
Comparison of results obtained on swatches and garments

Fabric type	Laundering conditions		Felting shrinkage W (%)	Felting shrinkage F (%)	Mussiness rating	Fuzz formation in laundering[1]
No. 82	Ten laundering and air drying cycles	skirt	4	2	—	—
		swatch	3	1	—	—
No. 83	Five laundering and tumble drying cycles	slacks	4	2	3.0	2.8
		swatch	8	2	3.0	1.6
Sport shirt fabric:						
Shrink-resistant-treated	Five laundering and air drying cycles	shirt	1.5–3	1–2	4.7	—
		swatches	2	2	4.5	1.2
	After five additional launderings and tumble drying cycles	shirt	2–4	1.5–3	5.7	1.9
		swatches	4	3	5.0	1.2
Untreated	Five laundering and air drying cycles	shirt	7–10	6–9	2.8	—
		swatches	10	11	3.8	1.8
	After five additional launderings and tumble drying cycles	shirt	10–16	12–16	2.0	2.1
		swatches	15	13	5.0	1.8

[1] A low value indicates little change due to laundering.

TABLE V
Effect of chlorination treatments on "wash-and-wear" characteristics of wool fabrics

Ten laundering and tumble dryng cycles

Fabric no.	Active chlorine (%)	Area felting shrinkage (%)	Mussiness rating	Fuzz formation in laundering: Visual rating[1]	Fuzz formation in laundering: Thickness increase (mils)
82	0	3	4.5	2.0	37
	2	0	4.2	1.1	22
	3	2	4.2	1.0	3
	4	2	3.8	0.6	4
129	0	13	3.2	1.0	35
	2	9	3.2	1.0	34
	3	5	3.8	0.7	19
	4	6	5.0	0.4	4
131	0	24	3.8	2.0	65
	2	10	4.5	1.7	51
	3	4	5.2	1.4	39
	4	4	5.7	1.2	39
113	0	36	2.3	1.4	49
	1	25	2.2	0.9	51
	2	19	2.3	1.3	46
	3	12	2.8	0.8	34
	4	5	3.8	0.8	27

[1] A low value indicates little change due to laundering.

not become stabilized in repeated launderings, but the rate of shrinkage decreased as the number of launderings increased.

There was no systematic effect of the number of launderings on mussiness ratings except in cases when cockling and puckering due to differential shrinkage of differently dyed yarns increased with increasing number of cycles. Comparison of the mussiness ratings of 25 fabrics taken after one, five, and 10 laundering cycles showed that the average range for any one fabric was 0.4 ratings; the largest range found was 1.3, the smallest 0 ratings.

The fuzziness increased, as expected, with increasing number of laundering cycles. However, ratings obtained at five and at 10 cycles generally ranked fabrics in the same order. Ten cycles appears thus to be a reasonable number for evaluation of "wash-and-wear" characteristics of wool fabrics.

COMPARISON OF SUBJECTIVE FUZZINESS RATINGS WITH COMPRESSOMETER MEASUREMENTS ——The subjective evaluation of the fuzz formation in laundering was generally based on the difference in the fuzziness rating of the laundered and the relaxed fabrics. Thus, a small number (a small fuzz increase due to laundering) is desirable. The Compressometer method also utilizes the difference in the thickness of the fuzz between laundered and relaxed fabric; obviously, the determination of what part of fabric thickness is due to fuzz and what part is due to the fabric body is somewhat difficult, and was arbitrarily made as described under "Methods—Fuzziness Rating" above.

Figure 4 shows that, for a large number of fabrics, only a rough correlation between these two measurements could be established. However, for any one fabric which underwent a variety of treatments, a good correlation was found, as shown in Table V and Figure 5. Similarly, for the two hard-finished fabrics, Nos. 55 and 129, described in Table II, increase in fuzziness measured visually (higher rating) corresponded to greater fuzz thickness determined by the Compressometer method; this did not, however, hold for the two flannels, Nos. 113 and 131. Thus, it appears that the Compressometer method cannot be applied to the comparison of fabrics which vary in construction and finish, and especially, to raised fabrics. It also seems to be affected by the mussiness and the shrinkage characteristics of the fabrics, and fails to differentiate between low, dense, and long, sparse fuzz. The Compressometer is, however, useful in the evaluation of the effect of chemical treatments on fuzz formation in laundering if fabrics with low shrinkage and mussiness characteristics are used for the experiments.

TYPICAL EFFECTS OF CHLORINATION TREATMENT —— The effect of laboratory chlorination treatments, varying in concentration from one to four percent active chlorine, is here chosen to exemplify the type of results obtainable by the above evaluation methods (Table V).

Increasing chlorine concentration did not improve the dimensional stability of Fabric 82, which had innately low shrinkage; while Fabric 113, a high-shrinkage fabric when untreated, reached reasonable stabilization at four percent active chlorine. Fabrics 129 and 131 seemed to reach equilibrium stabilization at three percent.

Increasing chlorine concentration had a beneficial effect on mussiness. Only Fabric 82, which was very smooth in the untreated state, seemed to get somewhat more mussy as the chlorine concentration increased. In the case of Fabric 113 there was an obvious reason for the better mussiness ratings of the stabilized fabrics, ie, less cockling as the shrinkage decreased.

The fuzz formation during laundering decreased roughly with increasing chlorine concentration (lower visual rating and lower thickness increase). This is also illustrated in Figure 5 where the more heavily shaded symbols (which correspond to higher chlorine levels) generally were found to correspond to lower fuzziness rankings more than the open symbols for untreated or lightly treated fabrics. Thus, chlorination generally controlled both shrinkage and fuzz formation during laundering. Again, Fabric 82 is noteworthy in this connection: while chlorination

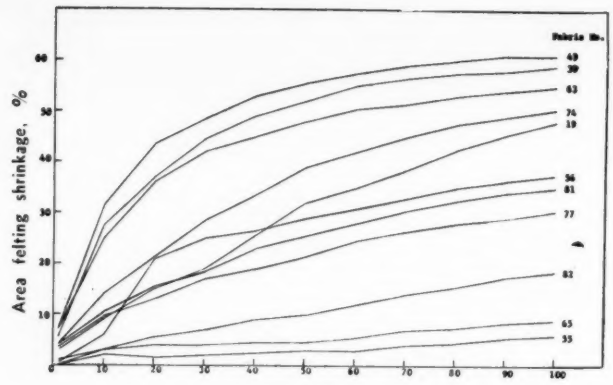


Figure 3
The effect of repeated laundering on area felting shrinkage of selected wool fabrics

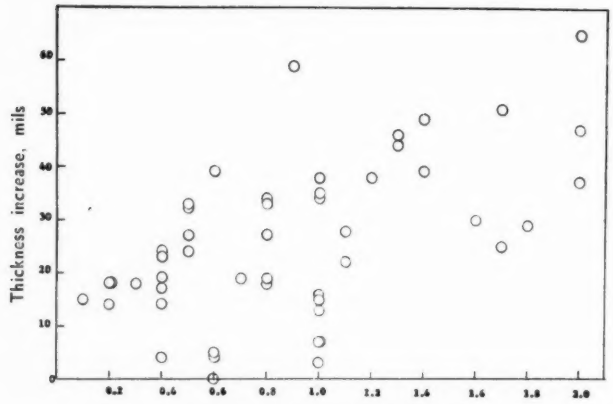


Figure 4
Fuzz formation in laundering: Visual rating vs thickness increase of various wool after 10 laundering and tumble drying cycles

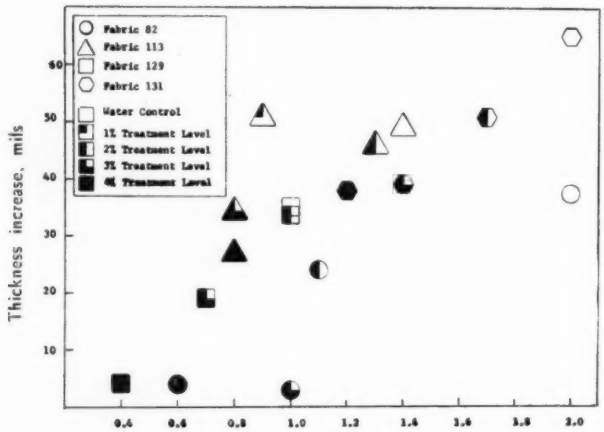


Figure 5
Fuzz formation in laundering: Visual rating vs thickness increase of four fabrics treated with various levels of active chlorine (10 laundering and tumble drying cycles)

did not improve its already good resistance to shrinkage, it was valuable in fuzz control. It thus appeared that the shrinkage of wool fabrics and at least part of the increase in fuzziness may have a common cause, the migration of fibers towards their root ends. Possibly any process which reduces fabric shrinkage will also go a long way to overcome wool's other pronounced shortcoming in the "wash-and-wear" field, fuzz formation. Work is under way in these laboratories on the evaluation of various treatments with this relationship in mind.

CONCLUSIONS

Wool, like other fibers, has its own set of advantages and disadvantages when used in "wash-and-wear" fabrics. Mussiness is less of a problem with wool than with cellulosics, especially when the fabrics are tumble dried. Shrinkage can be minimized and controlled by construction and/or stabilization treatments, and by the use of mild laundering conditions available in the modern washer. Fuzz formation in laundering is a problem somewhat unique to wool, though it is somewhat similar to pilling of synthetics; it, too, depends on construction, treatment, and laundering methods.

Of some significance is the finding that, among the untreated, commercial wool fabrics investigated, there were several which would be acceptable as judged by present "wash-and-wear" standards, even though no washability claims were made for these materials. If chemical stabilization treatments are applied, the range of such acceptable fabrics can be extended.

Work on the effect of fiber, yarn and fabric variables on the shrinkage, mussiness and fuzz formation of wool fabrics is in progress, and will be reported at a later date.

ACKNOWLEDGMENT

The sponsorship of this research program by the International Wool Secretariat and the Wool Bureau is gratefully acknowledged. The authors are also grateful to Gerald Laxer, of the Wool Bureau and A E Brown and J Menkart, of these laboratories, for many helpful suggestions.

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WRINKLE-RESISTANT COTTON WITH DURABLE CHLORINE RESISTANCE

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Finishing agents for producing wrinkle-resistant and "wash-and-wear" cotton fabrics have been developed. They consist of mixtures of dimethylol ethyleneurea and a formal of a polyhydroxy compound. These agents give good wrinkle resistance and chlorine resistance, both of which are durable to repeated launderings.

INTRODUCTION

BECAUSE of the importance that wrinkle-resistant and "wash-and-wear" finishes for cotton fabrics have attained, the search for improved finishing agents is being conducted intensively. Early finishes for these purposes had the property of retaining chlorine from hypochlorite bleaching solutions which subsequently resulted in strength loss or discoloration of the fabric. The widespread use of hypochlorite bleaching agents in the United States, even on dyed fabric (*4*), made the use of these finishing agents hazardous on all cotton fabrics and completely unacceptable on white goods.

To eliminate chlorine retention, newer types of finishing agents were developed, particularly the dimethylol derivatives of cyclic ureas. Ethyleneurea was one of the first cyclic ureas to achieve wide use for this purpose. Unfortunately, although dimethylol ethyleneurea finishes resist chlorine damage, the resistance is not durable. After repeated laundering, particularly under hot, alkaline conditions, the chlorine resistance of an ethyleneurea finish is lost, although a high degree of wrinkle resistance is retained. In addition, acidic conditions, such as too vigorous souring after laundering, will cause first loss of chlorine resistance, and then complete removal of the finish (*10*).

Two other types of cyclic urea compounds have been introduced since ethyleneurea. These are the triazones (*10, 11*) and the tetrahydropyrimidinones (*5*); the dimethylol derivatives of the former type are now widely used for the finishing of white cotton goods. Finishes derived from both of these types of compounds retain their chlorine resistance through alkaline launderings, and a triazone finish, in addition, has somewhat greater stability under acidic conditions than an ethyleneurea finish. Both of these agents, however, have objectionable features in undesirable side effects or additional cost when compared with ethyleneurea.

The blending of other reactive materials with dimethylol ethyleneurea has been employed to improve the durability of the chlorine resistance of ethyleneurea finishes. Epoxy compounds, for instance, have been used for this purpose (*3*). The present paper describes similar finishing agents which are mixtures of dimethylol ethyleneurea and formals of polyhydroxy compounds.

The use of formals of polyhydroxy compounds has been described for the dimensional stabilization of cotton and in some cases for wrinkle resistance treatments (*6*). Some compounds of this general type are available commercially. The formals used in this work give none or only low degrees of wrinkle resistance when applied alone to the fabric. Mixtures of these formals and dimethylol ethyleneurea, however, give a high degree of wrinkle resistance to cotton fabric. Fabric finished with these mixtures will have better chlorine resistance after laundering than fabric finished to the same degree of wrinkle resistance with dimethylol ethyleneurea alone. The acid resistance of the finish obtained with the mixture, unfortunately, is about the same as that of a dimethylol ethyleneurea finish.

MATERIALS AND METHODS

The dimethylol ethyleneurea used in this investigation was a commercial product obtained as a 50% solution. Several experiments repeated with dimethylol ethyleneurea synthesized in the laboratory gave results not significantly different from that obtained with the commercial product.

The two formals used in the investigation were laboratory preparations. One was a formal prepared from diethylene glycol and formaldehyde by a method described in the patent literature (*8*). The other formal was prepared from pentaerythritol and formaldehyde by the same procedure, substituting an equivalent amount of pentaerythritol for diethylene glycol.

Two metallic salt catalysts were used with these finishing agents, magnesium chloride and zinc nitrate as the hexahydrates. The latter salt was used with acetic acid added, in an amount equal to 10% of the weight of the zinc nitrate hydrate, to prevent precipitation of zinc in the treating bath.

Baths were prepared for the treatment of fabric samples by making aqueous solutions containing known percentages by weight of dimethylol ethyleneurea, formal, and catalyst. In some treatments a polyethylene emulsion was also added to give a known percentage of polyethylene in the bath.

The fabric used in this investigation was a desized, caustic-boiled and bleached, 80 x 80 cotton print cloth (3.2 oz/sq yd). Samples of this fabric were impregnated with the treating baths by means of pad rolls, using two dips and two nips, and a roll pressure to give approximately 75% wet add-on. The samples were mounted on pin frames at original dimension and dried for seven minutes at 60°C and cured immediately after for four minutes at 160°C except where another curing temperature is noted. Both drying and curing were conducted in ovens with air mechanically circulated. After curing, the samples were given the equivalent of a process wash in a home-type washing machine with a nonionic detergent.

After treatment, the weight add-on of finishing agent on the fabric samples was determined by comparing the initial and final weights of the air-equilibrated samples. The fabric samples were tested for crease recovery by the Monsanto method, tearing strength by the Elmendorf method, breaking strength on strips raveled to 80 threads, and moisture regain (*2*). Chlorine resistance was measured by the scorch test, AATCC tentative test method 69-1952 (*1*). Durability of the properties imparted to the fabric samples was determined by repeating the tests after five laundry cycles as described in AATCC Standard Test Method 14-1953 (*1*) with the samples being dried after each cycle.

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TABLE I

Properties of fabric treated with dimethylol ethyleneurea and diethylene glycol formal

Composition of treating bath			FABRIC PROPERTIES								
			Original					After five AATCC STM 14-1953 laundry cycles			
DMEU (%)	Formal (%)	Catalyst[1]	Add-on (%)	Crease recovery angle (degrees, W + F)	Tearing strength (gms, W + F)	Breaking strength (lbs, W)	Chlorine resistance[2]	Crease recovery angle (degrees, W + F)	Tearing strength (gms, W + F)	Breaking strength (lbs, W)	Chlorine resistance[2]
(Untreated fabric)			—	175	2026	51.1	97	185	1853	43.9	98
0	15	A	4.3	231	873	36.2	93	235	1027	33.5	97
0	15	B	2.7	233	853	—	—	—	—	—	—
7	0	A	3.1	274	834	28.5	94	270	880	27.1	42
7	0	C	3.6	275	920	31.2	108	283	1013	28.6	50
3	7	A	4.7	252	887	31.8	95	258	920	28.6	71
3	7	B	4.7	248	840	27.6	107	267	907	27.6	91
5	10	A	6.3	270	726	31.1	94	263	853	31.8	76
5	10	D	7.6	268	767	33.9	95	259	807	31.6	81
5	10	B	6.1	278	720	31.7	96	261	793	28.3	86
5	10	B[3]	7.1	279	760	28.3	100	273	873	26.7	96
5	10	C	6.3	274	794	31.9	98	265	880	32.7	76
7	10	A	7.8	269	734	30.4	103	265	880	28.1	76
7	10	B	9.0	283	766	27.9	106	297	780	29.1	96

[1] Catalyst: A, 6% $MgCl_2 \cdot 6H_2O$. B, 1% $Zn(NO_3)_2 \cdot 6H_2O$ and 0.1% acetic acid; C, 0.5% $Zn(NO_3)_2 \cdot 6H_2O$ and 0.05% acetic acid; D, 3% $MgCl_2 \cdot 6H_2O$.
[2] Percent of original strength retained in the scorch test.
[3] Cured at 150° C instead of 160° C.

TABLE II

Properties of fabric treated with dimethylol ethyleneurea and pentaerythritol formal

Composition of treating bath			FABRIC PROPERTIES								
			Original					After five AATCC STM 14-1953 laundry cycles			
DMEU (%)	Formal (%)	Catalyst[1]	Add-on (%)	Crease recovery angle (degrees, W + F)	Tearing strength (gms, W + F)	Breaking strength (lbs, W)	Chlorine resistance[2]	Crease recovery angle (degrees, W + F)	Tearing strength (gms, W + F)	Breaking strength (lbs, W)	Chlorine resistance[2]
(Untreated fabric)			—	175	2026	51.1	97	185	1835	43.9	98
0	15	A	1.0	167	1240	40.8	99	183	1167	41.0	81
0	15	B	0.3	186	913	36.2	94	191	1020	33.3	97
7	0	A	3.1	274	834	28.5	94	270	880	27.1	42
7	0	C	3.6	275	920	31.2	108	283	1013	28.6	50
5	5	A	3.8	259	820	31.2	93	264	873	34.0	20
5	5	B	3.9	267	753	32.0	95	271	800	28.5	81
7	5	A	5.0	265	793	30.1	85	260	834	31.6	23
7	5	B	5.2	276	793	29.4	103	271	826	29.2	92
7	5	B[3]	3.6	285	720	30.2	104	277	810	26.6	79
7	5	C	4.5	281	933	28.1	98	279	880	28.2	79
7	10	A	6.2	255	720	31.3	92	260	800	31.3	34
7	10	B	6.1	270	880	29.0	96	263	806	28.9	83
7	10	C	5.5	272	980	28.6	100	264	854	28.6	80

[1] Catalyst: A, 6% $MgCl_2 \cdot 6H_2O$; B, 1% $Zn(NO_3)_2 \cdot 6H_2O$ and 0.1% acetic acid; C, 0.5% $Zn(NO_3)_2 \cdot 6H_2O$ and 0.05% acetic acid.
[2] Percent of original strength retained in the scorch test.
[3] Cured at 150° C instead of 160° C.

RESULTS AND DISCUSSION

In Table I are given the results obtained on testing samples treated with mixtures of the diethylene glycol formal and dimethylol ethyleneurea. Table II shows the results obtained similarly with the mixtures of the pentaerythritol formal and dimethylol ethyleneurea. The data in both tables show the protective action of the formal by the strength retained in the scorch test after the fabric has been laundered. In most cases the crease recovery angles are reduced little or not at all by the use of formal in addition to dimethyol ethyleneurea, although a higher total add-on is apparently required. Tearing strengths are, in general, lower than that obtained with dimethylol ethyleneurea alone on this fabric, but are not reduced excessively. The moisture regains of the fabric samples, shown in Table III, are the usual for wrinkle-resistant cotton even with the higher add-on.

The data presented in the tables show that zinc nitrate is the better catalyst of the two used for these mixtures. With magnesium chloride, less durable chlorine resistance is obtained with either formal. In fact with the pentaerythritol formal and magnesium chloride catalyst, the mixture shows no improvement in chlorine resistance over dimethylol ethyleneurea used alone. Zinc nitrate with these mixtures, however, has a pronounced tendency to yellow the fabric on over-curing, and therefore curing conditions must be carefully controlled. Although none of the samples treated with mixtures and reported in the tables had noticeable yellowing, the samples treated with formals alone using the zinc nitrate catalyst were quite yellow. With the combination of zinc nitrate and the pentaerythritol formal mixture, it would probably be better to use the

TABLE III

Moisture regain of fabric treated with dimethylol ethyleneurea and formals

Composition of treating bath			Fabric properties		
DMEU (%)	Formal[1] (%)	Catalyst[2]	Add-on (%)	Crease recovery angle (degrees, W + F)	Moisture regain (%)
7	— 0	A	3.1	274	5.6
5	— 0	B	2.0	279	5.6
5	DG 10	A	6.3	270	5.7
5	DG 10	B	7.4	262	5.3
5	DG 10	B[3]	7.1	279	5.3
5	PF 5	B	3.9	267	6.1
7	PF 5	B	5.2	276	5.6
Untreated			—	—	6.6

[1] Formal: DG, diethylene glycol formal; PF, pentaerythritol formal.
[2] Catalyst: A, 6% $MgCl_2 \cdot 6H_2O$; B, 1% $Zn(NO_3)_2 \cdot 6H_2O$ and 0.1% acetic acid.
[3] Cured at 150° C instead of 160° C.

TABLE IV

Effect of polyethylene additive on the properties of fabric treated with dimethylol ethyleneurea and diethylene glycol formal

Composition of treating bath				Fabric properties			
				Original		After five AATCC STM 14-1953 laundry cycles	
DMEU (%)	Formal (%)	Catalyst[1]	Polyethylene (%)	Crease recovery angle (degrees, W + F)	Tearing strength (gms, W + F)	Crease recovery angle (degrees, W + F)	Tearing strength (gms, W + F)
5	10	A	—	256	734	265	873
5	10	A	1.5	276	1034	278	926
7	10	A	—	269	734	265	880
7	10	A	1.5	277	1000	278	1000
5	10	B	—	262	807	277	813
5	10	B	1.5	271	937	277	927

[1] Catalyst: A, 6% $MgCl_2 \cdot 6H_2O$; B, 1% $Zn(NO_3)_2 \cdot 6H_2O$ and 0.1% acetic acid.

TABLE V

Acid hydrolysis of dimethylol ethyleneurea—formal finishes

Composition of treating bath			Crease recovery (degrees, W + F) after 30 minutes at 40° C in solution of pH					Percent nitrogen after 30 minutes at 40° C in solution of pH				
DMEU (%)	Formal[1] (%)	Catalyst[2]	1.0	2.4	3.5	5.3	6.4	1.0	2.4	3.5	5.3	6.4
5	DG 10	A	179	198	222	253	256	0.08	0.24	0.57	0.79	0.76
5	DG 10	B	175	203	236	258	259	0.10	0.35	0.64	0.78	0.81
7	PF 10	B	162	194	241	262	269	0.12	0.49	0.93	1.13	1.12

[1] Formal: DG, diethylene glycol formal; PF, pentaerythritol formal.
[2] Catalyst: A, 6% $MgCl_2 \cdot 6H_2O$; B, 1% $Zn(NO_3)_2 \cdot 6H_2O$ and 0.1% acetic acid.

procedures in which a lower curing temperature, 150° C, or 0.5% zinc nitrate is employed. Both of these procedures produce satisfactory results, as shown by the tabulated data, and the lower catalyst concentration appears to give somewhat better tearing strength.

No tendency toward yellowing was observed when the magnesium chloride catalyst was used. However, as previously noted, this catalyst is only useful with the diethylene glycol formal.

The results obtained by using a polymeric softening agent with these mixtures are shown by the data in Table IV. These samples were treated with mixtures of dimethylol ethyleneurea and the diethylene glycol formal, with and without 1.5% of emulsified polyethylene in the treating bath. The polyethylene was obtained as a commercial product in an emulsion of 33% solids and added as such to give the desired concentration in the treating bath. A small amount of nonionic emulsifier was added to the commercial product to prevent coagulation during the treatment. Increases in both tearing strength and crease recovery were obtained by addition of polyethylene. Even more marked was the improvement in "wash-and-wear" properties of the fabric samples. All the samples noted in Table IV that were treated with a mixture containing polyethylene had superior "wash-and-wear" properties.

The acid resistance of the finish obtained from the dimethylol ethyleneurea and formal mixtures was tested by the method used by Mazzeno, Kullman, Reinhardt, and Reid (*9*). This method consists in measuring the crease recovery and nitrogen contents of a series of identically finished samples after subjecting them to solutions buffered to decreasing pH. The results obtained, Table V, show a drastic loss in nitrogen content occurs about pH 4 and is accompanied by a corresponding loss of crease recovery. This is similar to the result reported for the usual dimethylol ethyleneurea finish (*9*) and indicates poorer acid resistance than obtained with many other finishes (*10*). It is a distinct disadvantage of the dimethylol ethyleneurea finishing agent, either used alone or with the formals as described in this article. Even exposure to acidic solutions that do not destroy wrinkle resistance is likely to reduce chlorine resistance considerably.

Cotton articles, whether wrinkle-resistant or not, should not, of course, be submitted to such abusive laundering conditions as strong acidic souring. The American Institute of Laundering recommends a souring procedure for commercial laundering with a pH not below 5 (*7*). Unfortunately, excessively low pH under warm or hot conditions are used by many commercial laundries. In correct laundering practice the main hazard is the alkalinity of the wash liquors which causes the dimethylol ethyleneurea finish to become susceptible to chlorine damage. This defect is overcome by the use of formals, with dimethylol ethyleneurea as shown by resistance to the five AATCC STM 14-1953 wash tests. Home laundering is seldom, if ever, as severe as these tests. The finishes

described should, therefore, have satisfactory durability to all nonabusive laundering.

SUMMARY

Finishing cotton fabrics with mixtures of dimethylol ethyleneurea and formals of diethylene glycol or pentaerythritol produces fabrics with good wrinkle resistance. With an additive, such as polyethylene, a good "wash-and-wear" finish can be obtained. These finishes are chlorine resistant and retain their chlorine resistance through repeated alkaline laundering better than finishes obtained with dimethylol ethyleneurea alone. They still have the susceptibility of the dimethylol ethyleneurea finish to acidic conditions. This property renders it vulnerable to excessively hot or acidic souring conditions, but should not be a detrimental factor when normal souring procedures are followed.

ACKNOWLEDGMENTS

The authors wish to extend their thanks to the following for their assistance in testing the fabric samples in this investigation: Gloria A Gautreaux and members of the Product Evaluation Group of this Division, in particular, Julia M Sloan and Henry A Allen.

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DYEING OF WOOL AT SUBBOILING TEMPERATURES

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IN the course of the last five years, there has been considerable opportunity to study the dyeing behavior of many new dyestuffs of the Brilliant Alizarine Milling and the Lanasyn (neutral premetallized) types. In running various tests on their dyeing rate, it has been observed that many of them showed considerable exhaustion during the 60-minute temperature rise to the boil. Some showed complete exhaustion after this 60-minute preboil period.

It is a well-known fact that wool suffers more shrinkage, felting and general damage at the boil than at lower temperatures. Having noted that many of these newer colors were giving good color yield in the normal temperature-raising period, it was thought that these colors might offer the opportunity of dyeing wool at a temperature which would eliminate the problems involved when dyeing at the boil. On discussing the subject with the owners of two different high-quality yarn plants, it was found that the idea seemed to have considerable merit in that they were having trouble with certain types of yarns, especially cashmere. We also found a carpet mill interested in this idea, and have since learned they are dyeing much of their yarn at 190°F, using a number of the colors recommended.

One percent dyeings of different dyestuffs were made on worsted piece goods at temperatures of 165°F, 175°F and 185°F for one hour with sufficient ammonium sulfate to give a pH of 5.8. The figure of pH 5.8 was taken first, because it was an average optimum pH of many of the colors we had under consideration; second, the carpet mill with whom the subject was discussed had settled on this pH as the most suited for its work.

A series of dyeings were made of each color at the boil and at a pH of 5.8 at strengths of .40, .50, .60, .70, .80, .90 and 1.00%. By comparing the 1% dyeings of the colors at the various lower temperatures with the "step" dyeings at the boil, each color was given estimated value for the particular temperature at which it was dyed. This could also have been obtained by measuring the exhaust liquor on the spectrophotometer. However, with the system used, it has been possible to mount the tests on large cards in the fashion of a slide rule, which has proven to be most helpful in explaining the results to potential users of the idea.

The principal fastness requirements for material to be dyed by this method would be fastness to light and washing. Fade-Ometer and wash tests were made of the 20 colors dyed at each of the three temperatures. These were compared with dyeings of an equivalent strength dyed at the boil.

RESULTS

In most cases, dyeings made at either of the three lower temperatures gave brighter shades. Continued treatment at elevated temperatures yellows wool and causes a definite dulling of blues, violets and pinks.

In the table, it will be noted that the maximum value (1% strength) fastness to light and washing is obtained from a larger number of colors at 185°F than when dyeing at the lower temperatures. Good fastness is obtained with only slightly less color yield than at the boil.

The table indicates no appreciable loss in fastness to light or washing at 185°F with only occasional instances of lower fastness at 175°F. At 160°F, there is a fall off in fastness.

SUMMARY

It is believed that many of the colors in the tables and others now in process of testing can be dyed satisfactorily on carpet and knitting yarns. Special emphasis is placed on knitting yarns, cashmere or types easily felted or damaged by high temperature.

Some of the advantages are:

1) Saving of time
2) Saving of steam
3) Softer hand of yarn
4) Greater ease in winding and knitting
5) Further improvement in the results from cross dyeing. Cross dyeing with neutral (metallized) colors has been recommended because of their ability to dye at pH 5.5-7.0 (a condition favorable to cross dyeing), and their good yield at 180-185°F.

The strength values, as well as the effect on fastness, are combined in the table.

Effect of temperature on dyeing and fastness properties of dyestuffs

Dyeing Method:
All colors with exception of those with asterisk were dyed with: 2% ammonium sulfate (Liquor ratio 40:1) (pH at end of dyeing 7:1)

Those with asterisk were dyed with: 0.5% acetic acid (56%) 10% Glauber's salt (calc) (Liquor ratio 40:1) (pH at end of dyeing 5.8)

Color	Colour Index Designation	Percent exhaustion at lower temperatures (using 1% dyeing at 208-212°F as standard) 185° F	175° F	165° F	Shade difference (if any) at lower temperatures	Lightfastness (Fade-Ometer) using 1% dyeing at 208-212°F as standard 185°F	175°F	165°F	Washfastness (140°F—one hour) using 1% dyeing at 208°-212°F as standard 185°F	175°F	165°F
Lanasyn Brilliant Yellow 5GL	—	83	83	75	trace brighter						
" Yellow 2RL Pat	CI Acid Yellow 80	100	80	60		equal	equal	sl poorer	equal	sl poorer	sl poorer
" Yellow GL	CI Acid Yellow 114	60	55	40		trace better	trace better	sl better	trace poorer	trace poorer	X
" Yellow 2GL	—	75	65	60	slightly redder	equal	equal	equal	equal		X
" Yellow 3GL Pat	CI Acid Yellow 111	90	78	73	trace brighter	equal	equal	equal	sl poorer	poorer	consid poorer
" Yellow GLN Pat	CI Acid Yellow 112	85	78	65	slightly redder	superior	consid superior	consid superior	trace poorer	poorer	consid poorer
" Orange RL	CI Acid Orange 86	70	65	60	slightly redder	equal	equal	equal	equal	sl poorer	sl poorer
" Orange RLN paf	CI Acid Orange 82	85	70	55	slightly brighter	equal	equal	superior	trace poorer	sl poorer	sl poorer
" Bordeaux RL Pat	—	95	85	73	slightly yellower & trace brighter	trace better	trace better	trace better	equal		X
" Bordeaux GRL	CI Acid Red 213	85	75	60	equal	equal	equal	equal	sl poorer	poorer	consid poorer
" Brilliant Red 2BL paf	—	90	85	80	trace brighter	sl superior	sl superior	sl superior	sl poorer	poorer	consid poorer
" Red BL paf	CI Acid Red 215	90	75	60	equal	sl superior	sl superior	equal	sl poorer	poorer	poorer
" Red 2GL Pat	CI Acid Red 216	90	60	55	slightly bluer	equal	equal	equal	sl poorer	poorer	poorer
" Brilliant Green BL Pat	CI Acid Green 40	90	80	75		sl inferior	equal	equal	sl poorer	poorer	consid poorer
" Brown GL	—	95	80	60	equal	equal	equal	equal	trace better	equal	X
" Brown GRL Pat	—	95	75	60	at 185°F, equal; at 175° & 165°F, slightly duller	superior	equal	equal	poorer	consid poorer	consid poorer
" Brown RL paf	CI Acid Brown 28	80	70	60	slightly brighter & redder	sl superior	sl superior	consid superior	equal	poorer	poorer
" Brown 2RL	CI Acid Brown 45	85	65	60	redder & trace brighter	equal	equal	equal	trace better		X
" Brown 3RL paf	—	85	65	55	very much redder (checked)	equal	equal	equal	sl poorer	poorer	poorer
" Grey BL	CI Acid Black 58	95	85	75	brighter and more violet	consid superior	superior	sl superior	trace poorer	sl poorer	sl poorer
" Grey BLR	—	95	75	60	trace redder & slightly brighter	trace better	trace better	trace better	equal	equal	X
Brilliant Alizarine Milling Green 2GL Pat	—	85	70	50	bluer & trace duller	equal	equal	equal	equal	equal	consid poorer
Brilliant Alizarine Milling Blue G	CI Acid Blue 127	85	80	70	slightly brighter	superior	sl superior	sl superior	equal	equal	equal
Brilliant Alizarine Milling Blue FGL Pat	—	90	75	55	trace brighter	equal	equal	sl poorer	trace poorer	trace poorer	X
Brilliant Alizarine Milling Red FBL Pat	CI Acid Red 143	75	70	60	slightly bluer & trace brighter	equal	equal	equal	equal		X
Brilliant Alizarine Milling Violet FBL Pat	CI Acid Violet 48	85	70	65	trace brighter	equal	equal	sl poorer	trace poorer	sl poorer	poorer
Xylene Milling Yellow 3GL	CI Acid Yellow 75	90	80	75		equal	equal	equal	equal		X
" Fast Rubine 3GP Pat	CI Acid Red 57	50	45	40	slightly brighter	equal	equal	equal	equal	equal	equal
Alizarine Light Brown BL	CI Acid Brown 36	90	80	70	brighter & redder	equal	trace poorer	trace poorer	equal		X
Xylene Fast Yellow P*	CI Acid Yellow 61	95	85	85		equal	equal	equal	equal	sl poorer	X
" Fast Orange P paf*	—	95	85	75	slightly brighter & redder	equal	equal	equal	equal	equal	equal
" Fast Red P*	CI Acid Red 118	90	90	85	slightly brighter & redder	trace better	poorer	poorer	equal	equal	X
" Fast Bordeaux P*	—	95	85	85	brighter & redder	equal	equal	equal	trace better	sl poorer	poorer
" Fast Blue P*	CI Acid Blue 82	100	90	85	brighter & redder	equal	equal	equal	trace poorer	equal	equal
" Fast Grey P*	CI Acid Black 50	100	95	85	brighter & bluer	equal	equal	trace poorer	equal	trace poorer	trace poorer
Brilliant Alizarine Sky Blue BS	CI Acid Blue 129	95	90	80	slightly brighter	equal	equal	equal	equal	sl poorer	poorer
										equal	equal

X Because of poor exhaustion no test made

A BREAKTHROUGH IN PIGMENT TECHNOLOGY

— The New Yellow-Green Phthalocyanine —

EMIL A WICH

Dyestuff & Chemical Div
General Aniline & Film Corp
New York, NY

A new phthalocyanine pigment has been introduced by the General Aniline & Film Corporation which extends the color spectrum of the phthalocyanines to yellow-green shades. The properties of the new pigment are very similar to those of phthalocyanine green. It is lightfast, chemical resistant, and heat stable. It may be used for textile printing and padding of all types of fabrics. There is almost no limit to the systems with which it can be used, or to the aftertreatment which may be employed. A water dispersion of very fine particles is available for use in spin dyeing. Water-dispersible paste and presscake are also available for preparing concentrates and textile printing and padding formulations.

PHTHALOCYANINE pigments are an important factor in nearly all phases of the color industry. They are used in paints, inks, plastics, rubber, paper and textiles. The textile industry has favored these remarkable pigments for many years because of their brilliant color, excellent fastness to light, heat stability, and resistance to bleeding and to degradation by acids and alkalis.

Unfortunately, these highly desirable properties have been limited to the narrow color range marked by phthalocyanine blue, which is very nearly a pure blue, and phthalocyanine green, a bright green pigment with a blue hue. Many companies have devoted years of research to the problem of synthesizing new phthalocyanine compounds which would expand this color spectrum. However, the problem resisted solution by the best pigment chemists for over 20 years, and it was not until this year that the long search was finally rewarded.

The reward was a new phthalocyanine pigment having a clean, bright green color with a distinct yellow hue. It was developed by the Dyestuff & Chemical Division of General Aniline & Film Corporation and has been designated by them as Heliogen* Viridine Y. The new pigment is already in production and is commercially available.

Quite appropriately, in view of its "Kelly Green" color, the new pigment made its first "public appearance" in the green stripe which runs down the middle of New York's Fifth Avenue to mark the route of the traditional St Patrick's Day parade.

COLOR —— The reflectance-transmittance curve in Figure 1 shows the color response of Heliogen Viridine Y in a transparent sheet of cellulose acetate containing 0.5% of the pigment. Figure 2 shows the color response of phthalocyanine green (CI Pigment Green 7) and benzidine yellow (CI Pigment Yellow 12) blended to produce a similar color.

The color values for the viridine pigment are shown in Table I, which also lists the values for phthalocyanine green for comparison.

* Reg US Pat Off by General Aniline & Film Corp.

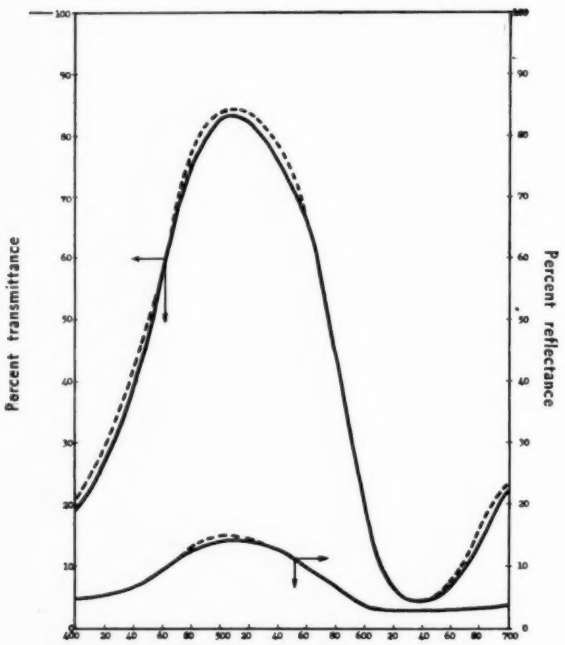


Figure 1
Heliogen Viridine Y
——unexposed; — — — exposed 185 hours

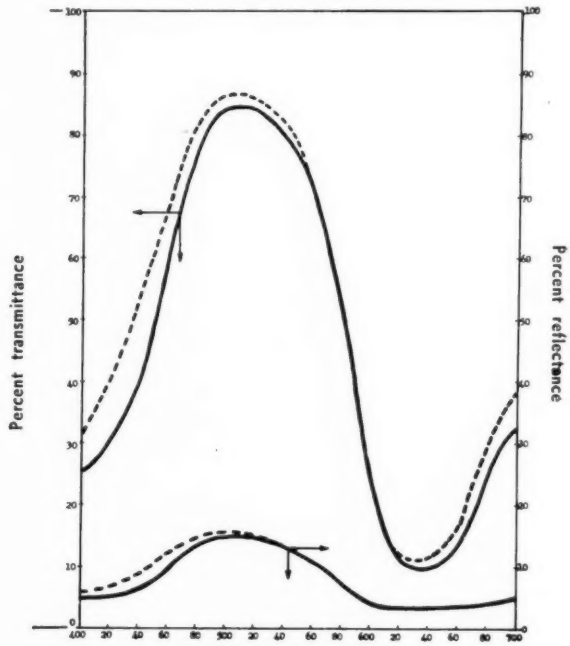


Figure 2
96.5% CI Pigment Green 7
3.5% CI Pigment Yellow 12
——unexposed; — — — exposed 185 hours

LIGHTFASTNESS——The lightfastness of the viridine pigment is graphically illustrated by the dotted lines in Figure 1 and a comparison of the response of the blend shown in Figure 2. The dotted lines show the color response of the pigments after exposing the plastic samples in a Fade-Ometer for 185 hours. The color response for the viridine pigment is virtually unchanged. The phthalocyanine green-benzidine yellow blend, however, has become more blue, approaching the color of phthalocyanine green by itself. This is due to the fading, or loss of tinctoral strength, of the yellow pigment, which is indicated by the increase in both the transmittance and reflectance of blue light (440-460 mμ) in the curves.

PHYSICAL PROPERTIES OF THE NEW PIGMENT——The results of extensive testing show the properties of the new yellow-green pigment to be almost identical to those of the well-known and widely used phthalocyanine green. It has the same excellent lightfastness, resistance to acids and alkalis, and heat stability for which phthalocyanine green is noted.

Table II presents further physical and fastness properties of the new pigment.

Tests made specifically to determine the suitability of the pigment for use in textile prints have shown excellent results. Textile prints showed no signs of fading after 100 hours Fade-Ometer exposure. The washfastness, crockfastness, chlorine fastness and drycleaning fastness of textile prints made with the new pigment were all found to be excellent.

FORMS AVAILABLE——The new pigment is available in toner form, presscake, and water dispersions both anionic and nonionic. Data on the toner has been given above.

The presscake is furnished at approximately 35% solids. It has a pH of 7.3 and is easily dispersed. It may be used in either oil or water textile printing systems.

Heliogen Viridine Y Supra Paste is an anionic water dispersion of very fine pigment particles (one micron or less in diameter). The dispersion contains 20% pigment, has a solids content of 30%, and a pH of 10-11.

TABLE I
Color values of Heliogen Viridine Y and phthalocyanine green

	Heliogen Viridine Y	*Phthalocyanine green*[1]
Tristimulus values:		
X	.2846	.2640
Y	.4271	.3926
Z	.4739	.5077
Chromatic coordinates:		
x	.2400	.2267
y	.3602	.3372
Munsell renotation:		
Hue	1.25 BG	4.71 BG
Value	6.98	6.73
Chroma	8.8	8.73

Spectrophotometer results on five parts pigment: 95 parts TiO_2[2] using $MgCO_3$ sphere.

[1] CI Pigment Green 7 (CI 74260).
[2] Rutile titanium dioxide.

TABLE II
Physical and fastness properties of Heliogen Viridine Y Powder

Specific gravity	2.4
Weight/gallon	19.99
Bulking value (gal/lb)	0.05003
Oil absorption number	30.
pH	7.7
Resistance to solvent bleed	
Alcohol	no bleed
Xylene	no bleed
Lacquer thinner	no bleed
MEK	no bleed
Mineral spirits	no bleed
Diethylene glycol	no bleed
Acid and alkali stability	
HCl (5%)	excellent
NaOH (2%)	excellent
Acetic acid (2%)	excellent
Heat stability	
One-half hour at 350° F	excellent

TABLE III
Fastness properties in spin dyeings (0.5%)

Light fastness (Fade-Ometer) 200 hours	excellent
Washfastness	excellent
Chlorine fastness	excellent
Stability in viscose dope	very good (No change after 7 days storage at 25° C)

APPLICATION——The Heliogen Viridine Y pigment is extremely easy to use and apply. Because of its superior chemical resistance and heat stability, there is no practical limitation on the wetting agents, binders, solvents, or aftertreatment that may be employed. Furthermore, the blending of pigments may be eliminated in many cases where a pure yellow-green color is desired. The greatest advantage offered by the new pigment is lightfastness. Yellow-green prints and padded fabrics no longer need fade and revert to a blue-green after exposure to sunlight.

TEXTILE PRINTING & PADDING——Either oil-in-water or water-in-oil systems may be prepared from the presscake. The materials and methods employed in dispersing the pigment and preparing the concentrate are the same as those used for other pigments in general, and phthalocyanine green in particular. The pigment may be used for blending with other pigments to obtain various shades and depths.

Pigment concentrates for textile printing will generally be prepared at about 15% pigment content. They may be cut from 1:4 to 1:100 with suitable binder and applied without loss of the fastness properties of the pigment.

Pigment padding formulations may be prepared either from the oil-in-water concentrates supplied by color compounders, or by mixing the anionic water dispersion with a suitable binder. The pigment concentration used for padding is usually in the range of from 0.5% to 3%.

SPIN DYEING——One of the basic requirements for pigments used in spin dyeing is fine particle size. Pigment particles large enough to block the narrow orifices of the spinnerettes used in the manufacture of viscose rayon must be avoided. The Supra Paste is specially conditioned to reduce the particle size of at least 99% of the pigment particles to one micron or less, so that it may be used in this process. Pigment concentrations of as little as 0.5% may be used to impart bright, clear colors in the lighter shades. Here again, eliminating the blending and mixing of pigments may allow greater economy in operation and permit more precise color control in processing.

Table III presents the results of tests on viscose rayon that has been colored by spin dyeing with 0.5% of the viridine pigment.

COST——The cost of the new yellow-green pigment is the same as that of phthalocyanine green. The price structure for the various forms in which it is available is similar to that of the corresponding phthalocyanine green types.

FUTURE PAPERS

The following papers will appear shortly in *American Dyestuff Reporter:*

"An Investigation of Inorganic Salts and Oxides as Finishes for Cotton"
"The Chemistry of the Aminonaphthoquinones"
"Durable Creases Produced in Cotton and Resin-finished Cotton Fabrics by an Alkali Process"
"The Improvement of Selected Physical Properties of Partially Acetylated Cotton Fabrics"

• PATENT DIGEST •

PAUL WENGRAF

DYEING WOOL WITH ANTHRAQUINONE VAT DYES ——— Strictly controlled temperature and pH C,4,02

USPat 2,857,235 (Secy of Army——Coscia——Oct 21, 1958)

The preamble to this patent refers to U S Pat 2,508,203 (Allied Chemical & Dye—Weber—May 16, 1950), which protects a process for dyeing wool with anthraquinone vat dyes at pH values of 12 or 13 within dyeing times of 15 to 60 minutes. Reportedly, the general opinion of dyeing experts is that the alkalinity in the dyeing bath must not exceed pH 12.5 (when measured electrometrically) if wool damage is to be avoided.

It has been found that the alkalinity can be increased to approx 12.8-13.1, provided temperature conditions and dyeing times are carefully regulated. At higher alkaline values, the affinity of dye to wool is substantially increased; thus a deeper shade is obtained in very short times of contact, limiting the risk of damage to the wool structure. For example, at pH 12.9± 0.1 a contact time of 20-40 seconds is preferred; while at pH of 13.1, dyeing in 10 seconds is advisable. It is important to neutralize the fibers immediately after contact with the leucovat. It is also suggested that a protecting agent, such as ethylene dibromide, be added under these conditions.

Temperature of the dyebath must be adjusted within 120-160°F. At lower temperatures the adsorption of the leucovat is slow, while temperatures above 160°F might cause wool damage. It has been observed that the temperature of the dyebath adjusted to the higher pH range (13.0-13.1) should be between 120-155°F.

Dyeing is carried out in a hydrosulfite vat as usual and the oxidizing bath containing acetic acid and ammonium persulfate is kept at 75°F. The process reportedly is most suitable for the dyeing of wool tops. Tightly woven fabrics may be insufficiently penetrated in the short dyeing time.

Among the references cited by the Patent Office:

U S Pat 2,508,203 (above). [Cf *Am Dyestuff Reptr 39,* 765 (1950)]

U S Pat 2,508,714 (Harris Research Labs/1950): increasing the resistance of wool to attack by oxidizing, reducing, or alkaline agents by reacting it with alkylene dihalides in the presence of a reducing agent. The ruptured disulfide linkages are rebuilt in a more stable form. [Cf *Am Dyestuff Reptr 39,* 726 (1950)].

ABRASION RESISTANCE OF RESIN-FINISHED FABRICS IMPROVED ——— Superheated steam introduced in curing G,2,04

USPat 2,859,136 (Tootal Broadhurst Lee Co——Marsh, Blease——Nov 4, 1958)

Textile fabrics are known to acquire increased shrink and crease resistance when certain resin finishes are applied. These desirable effects are counterbalanced, however, by a loss in abrasion resistance, according to the current specification. Hitherto it has been necessary to accept a compromise. In cases where abrasion resistance was particularly important, smaller amounts of resins had to be incorporated and full crease- or shrinkproofing effects had to be sacrificed.

The object of the present patent is to reduce the loss in abrasion resistance in textile fabrics composed of regenerated cellulose, while at the same time incorporating large quantities of urea- and/or melamine-formaldehyde condensates. This reportedly has been accomplished by introducing into the baking oven, under atmospheric pressure, a large amount (at least 50% of the oven's volume) of superheated steam. It has been suggested in the past that an atmosphere of steam be maintained during curing, but the content of steam supplied under a pressure of 40 psi in customary curing chambers amounted to less than 20%, according to exact measurements.

A table (col 4, lines 5-23) demonstrates the improved abrasion resistance in correlation with the increased steam content. Quite generally the improvement was found to be striking when the atmosphere contained at least 50% steam. Testing was done on the "ring-wear-testing machine" described in *J Textile Inst 26,* p 93 (1935).

Example: Urea and neutral formaldehyde mixed with some ammonia were refluxed and cooled, and $(NH_4)H_2PO_4$ was added as a catalyst. Two samples of viscose fabric were impregnated in this solution, squeezed to a 100% wet pickup, and dried at 40°C. One of the samples was dry cured at 150°C, as usual; the other was cured at the same temperature in an atmosphere containing 16 pbv air and 84 pbv steam. Both samples were then tested on the "ring-wear-testing machine." The dry-cured samples wore through at 1148 cycles; the steam-cured sample, at 2162 cycles.

Among the references cited by the Patent Office:

U S Pat 2,088,227 (Tootal Broadhurst Lee/1937): improving the crease resistance of textiles by a treatment with methylolurea plus a potentially acid salt, followed by curing.

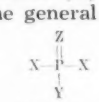
U S Pat 2,484,598 (Alrose Chem Co/1949): rendering cellulosic textiles crease-resistant by impregnating with a catalyst-free resin precondensate and curing in steam which contains volatile organic acid at 212-220°F up to 250°F. This reference, however, does not mention injection of superheated steam of more than 50% of the chamber's volume at atmospheric pressure. [Cf *Am Dyestuff Reptr 39,* 97 (1950)].

FLAMEPROOFING OF TEXTILES with polymers of ethylene imine-phosphorus derivatives and amines G,2,03

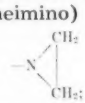
USPat 2,859,134 (Secy Agric——Reeves et al ——Nov 4, 1958)

New P-N-containing polymers are prepared by reacting an aziridinylphosphine oxide or sulfide, subsequently termed as "APO" or "APS" respectively (A), with an amino compound having a plurality of H atoms and/or CH_2OH radicals attached to trivalent N atoms (B).

"A" has the general formula



wherein X and Y stand for the azirinidyl (ethyleneimino) radical



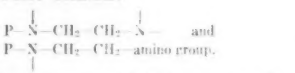
optionally Y may also be a dialkylamino group; Z is oxygen in APO and sulfur in APS. An example is $[N(CH_2)_2]_3PO$ tris(aziridinyl) phosphine oxide.

"B" may be urea or melamine, ethylamine, ethylene diamine, amide, and so on (col 2, lines 20-32).

Polymers of A and B used in the

current invention are prepared by gently heating an aqueous solution of APO or APS with the amino compound. Fibrous materials are impregnated with the aqueous solution of the combined monomers or with a dispersion of the partly polymerized compounds and then cured, following which the combustibility of textiles is reported to be durably reduced.

These polymers are found to contain the recurrent chains:



Example: A cotton fabric was padded in an aqueous solution of 15 pbw APO and 5.2 pbw ethylene diamine (equimolecular amounts) in 47.1 p water. The pH of the solution was 12.1. The fabric was thereupon dried for 15 minutes at 90° C and cured for five minutes at 140° C. After a thorough washing, it contained 13.4% of a resin and proved to be highly flame resistant while hand and tensile strength appeared to be substantially unaltered.

Among the references cited by the Patent Office:

U S Pat 2,781,281 (Ciba/1957): rendering fabrics flame-resistant by applying a reaction mixture obtained from a urea-phosphoric acid condensate and a methylolmelamine. [Cf *Am Dyestuff Reptr 46*, 928 (1957)].

U S Pat 2,672,459 (Am Cyanamid Co/1954) describes N-N′-dialkyleneimides of thiophosphonic acid, which are polymerizable to plastics and useful as new textile chemicals by reason of the reactive ethylene-imino group.

ORGANOSILOXANE EMULSIONS readily absorbed by substrates G,2,02

USPat 2,842,509 (Dow Corning Corp——Shannon ——July 8, 1958)

Organosilicon emulsions used for hydrophobing of textiles are generally applied to fabrics by immersion, drying and curing. According to the preamble to this patent, this method has proven to be satisfactory in continuous operations only. It is not suitable for batch operations because, in this event, the bath is not completely absorbed and a great amount of the siloxanes is wasted due to gelling of the emulsions in the long-standing bath. Thus a siloxane emulsion that is preferentially adsorbed by the fabrics would be desirable.

This reportedly has been accomplished by applying a siloxane emulsion containing a nonionic emulsifier and from two to 80% (bw of the siloxane content) of a polyimine or a quaternary ammonium polyglycol as substantive agents. Any emulsifiable siloxane may be employed, ranging from viscous resins to thin fluids (see col 2, line 66 to col 3, line 34). They may be emulsified with polyalkylene glycols, esters of polyhydric alcohols of nonionic character, etc (col 4, lines 7-18).

The substantive agents can be polyethylene imines derived from ethylene diamine and propylene diamine, or they may also be used in the form of their hydrohalides, such as

$$Cl(CH_2CH_2NH)_4CH_2CH_2Cl$$

or other salts. Polyglycol quarternary ammonium compounds suitable as substantive agents are prepared from halogenated polypropylene glycols by reaction with a tertiary aliphatic amine (col 3, line 35 to col 4, line 6).

Suitable catalysts for setting the siloxanes are water-soluble titanates, zirconates, sodium acetate, zinc acetate, or the like. Alkanolamine titanates are preferred for wool treatment.

This treatment is said to be suitable for any type of fabric, including those made from inorganic fibers and natural or synthetic fibers of any kind.

Example: A mixture of resins, composed of monomethylsiloxane, monophenylsiloxane and diphenylsiloxane, was emulsified with an alkylether of polyethylene glycol. The substantive agent used in this example was a polyimine of constitution

$$H_2N(CH_2CH_2NH)_3H.$$

The siloxane content of this emulsion reportedly was deposited completely on a cotton sample within 60 minutes of immersion and agitation.

Another example (#4) refers to a polyglycol quaternary ammonium compound as substantive agent.

No references are cited by the Patent Office.

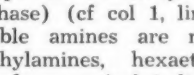
PIGMENT PRINTING PASTE——— Reaction products of epichlorohydrin with ammonia or amines as fixing agents D,2,07

USPat 2,844,490 (Farbenfabr Bayer——Lehmann et al——July 22, 1958)

The preamble refers to copending U S Patents 2,779,686 [cf *Am Dyestuff Reptr 46*, 696 (1957)] and 2,800,417, which describe the fixation of pigments by N-containing basic polymers that bear in the same molecule groups capable of crosslinking with the amino groups at elevated temperature.

According to the current patent, other fixing agents of similar characteristics contain free epoxy groups beside the basic groups; these agents are prepared from epichlorohydrin and ammonia, amines or polyamines. It has been observed that compounds of this type self link by reaction of the free epoxy groups with basic amino groups upon being heated to 80-200°C. Presumably, diamino hydroxypropane and mixtures of higher polyamines are formed from ammonia and epichlorohydrin in the first phase, while the amine mixtures condense further with epichlorohydrin to products containing free epoxy groups. In starting with polyamines, compounds containing such free epoxy groups result directly (this in the first phase) (cf col 1, lines 44-61). Suitable amines are methylamine, ethylamines, hexaethylene, diamine, and so on (col 1, lines 64-70).

Example: One mol epichlorohydrin was added within a half hour to an aqueous solution of ⅛ mol diaminopropyl methylamine



at 55°C. After this was stirred for approx four hours, the viscosity had risen to 200 centipoise; thereupon, concentrated hydrochloric acid was added to stabilize the solution. Seventy p of this viscous liquid were stirred with 6 p of a 15% copper phthalocyanine paste and 924 p water. A fabric padded in this composition and heated to 120°C for five minutes showed a coloration of excellent fastness to rubbing and washing.

Among the references cited by the Patent Office:

U S Pat 1,977,251 (Du Pont/1934): reaction products of epichlorohydrin and ammonia reportedly are efficient assistants in printing with vat dyes (Indanthrenes).

U S Pat 2,601,661 (Am Cyanamid Co/1952) describes a colored oil-in-water emulsion, the inner phase of which consists of a water-immiscible liquid; the continuous phase consists of an amino-formaldehyde resin plus a thermoplastic, acid-stable colloid, eg, a solution of methylcellulose in water. [Cf *Am Dyestuff Reptr 42*, 51 (1953)].

U S Pat 2,643,958 (Farbenfabr Bayer/1953) describes fixing agents for textile printing pastes. These are characterized as combinations of polymerizable amines (among them the reaction product of ammonia plus epichlorohydrin—see col 6, lines 20-21) with crosslinking agents, such as addition products of diketones and diisocyanates. These "polyisocyanate formers" are capable of reacting with primary or secondary amino groups. [Cf *Am Dyestuff Reptr 43*, 17 (1954)].

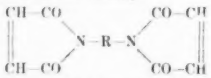
STABILIZING OF WOOL——— Crosslinking of thiol groups by cyclic unsaturated compounds A,2,04

USPat 2,850,351 (Secy of Agric——Moore, Pardo Jr——Sept 2, 1958)

Keratinous materials, particularly wool, hair, etc, are stabilized accord-

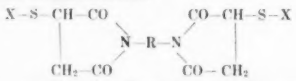
ing to known methods by replacing the easily attacked —S—S— linkages with alkylene groups or the like. The disulfide linkage is disrupted primarily by reduction to SH (thiol) groups, which are relinked in a subsequent treatment, eg, with ethylene dihalide, to form the more stable group —S—CH_2—CH_2—S. The current specification states that relinking with alkylene dihalides requires a strong alkaline medium for binding the hydrohalic acid. The alkali might have a weakening effect on the keratinous fibers. [See the method described in U S Pats 2,508,713-4 of Harris Res Labs; abstracted in *Am Dyestuff Reptr 39*, 726 (1950)].

According to the present patent, relinking of the thiol groups is accomplished with compounds which bear an acyl-nitrogen group, wherein each acyl radicle contains an olefinic linkage. Typical compounds are diimides of maleic acid,



wherein R stands for any hydrocarbon chain. A great number of diimides, diamides, and carbamide derivatives corresponding to the above formula is enumerated in the specification (col 2, line 54 to col 6, line 42).

The crosslinking agents called for in the current method act, in contrast with former methods, by simply adding the polypeptide chains that bear a thiol group (X—SH) to each side of the crosslinking agent of the above formula, thus forming relinked chains of the constitution



Compounds having two CO groups adjacent to the unsaturated linkage are preferred by reason of increased activation of the double bonds.

An example describes the primary reduction and following crosslinking process with five different agents (substituted dimaleimides, bisacryl- and bis-methacryl amides) in a closed vessel at 25°C for 30 hours. The suspension of the crosslinking agent is buffered at pH 8.0.

Among the references cited by the Patent Office:

U S Pat 2,615,782-3 (Procter & Gamble/1952): keratinous material, pretreated with thioglycollate to disrupt the disulfide linkages by reduction, is reacted with an organic thio (or dithio) sulfuric acid salt (lauryl thiosulfate) to restore at least part of the —S—S— bonds.

U S Pat 2,739,033 (Du Pont/1956): keratinous materials in which the disulfide linkages have been broken by reduction are reacted with an alkaline solution of a dihalo dicarboxylic acid.

WATER-REPELLENT COMPOSITION ——— Carbamide-formaldehyde condensate, higher amides, taurinating agents

G,2,02

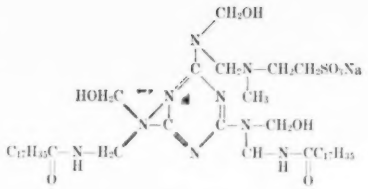
USPat 2,861,054 (Montclair Res Corp——Ellis Foster Co——Rust, Canfield——Nov 18, 1958)

Water-dispersible compositions, which impart durable water-repellent effects upon curing, are obtained, according to the current method, by reacting a compound of the carbamide group (A) with formaldehyde, a higher fatty acid amide (B), and finally with a taurinating agent (C).

Component A may be urea, thiourea, melamine, guanidine, and derivatives thereof. By reaction with formaldehyde, the corresponding methylol derivatives are obtained as usual.

Component B may be stearamide or any other fatty acid amide having a chain length of nine or more C atoms (preferably 18-24 C).

The "taurinating agent" (Component C) may be the sodium salt of tauric acid itself, $NH_2CH_2CH_2SO_3Na$, or any derivative thereof. N-methyltaurine is preferred (col 2, lines 21-51). A final, water-dispersible product, made by first condensing hexamethylolmelamine with stearamide, then with the Na salt of N-methyltaurine, has the formula



Upon curing in the presence of an acid catalyst, the taurine group may be split off or at least the water-dispersing effect of the group may be overcome so that the resin is insolubilized within the fibers (col 3, lines 42-48). A water-repellent effect, resistant to soaping and drycleaning, is reported.

Example: The condensate of A plus B was obtained by heating a mixture of melamine, p-formaldehyde, stearamide, and wax in a solvent solution of morpholine and propanol to 80-90°C for three hours. An aqueous solution of N-methyltaurine was slowly stirred in at the same temperature. The resulting water-dispersible condensate was homogenized into a smooth paste and diluted with water to 5% solids content. After the sample was padded at 25°C, dried at 60°C, and cured at 150-155°C, its spray-rating was found to be 100 and resistant to two hours' laundering. (See table on col 5, lines 37-47).

Among the references cited by the Patent Office:

U S Pat 2,377,868 (Gen Electric /1945) refers to synthetic materials (moldable compositions) produced by condensing an aminotriazine with formaldehyde and an aminocarboxylic acid (glycine in the examples).

U S Pat 2,537,667 (Monsanto Chem Co/1951): a dispersion of distearoyldiamino methane, a solvent-soluble butylether of tetranethylolmelamine, and paraffin wax is used to waterproof fibrous materials.

Brit Pat 628,818 (Am Cyanamid/1949) describes the reaction of methylolmelamine with an aminocarboxylic or aminosulfonic acid, eg, sodium taurinate (ex #19), to obtain leather tanning, bleaching and filling agents.

General Calendar

AMERICAN OIL CHEMISTS SOCIETY
Sept 28-30 (Statler Hotel, Los Angeles, Calif)

AMERICAN TEXTILE MACHINERY ASSOCIATION
May 23-27, 1960 (American Textile Machinery Exhibition, Auditorium, Atlantic City, NJ)

ASSOCIAZIONE COSTRUTTORI ITALIANI DI MACCHINARIO PER L'INDUSTRIA TESSILE
September 12-21 (3rd International Textile Machinery Exhibition, Milan Fair, Milan, Italy)

DRUG, CHEMICAL AND ALLIED TRADES SECTION, NY BOARD OF TRADE
Sept 17-20 (69th Annual Meeting, Sagamore Hotel, Bolton Landing, Lake George, NY)

INSTITUTE OF TEXTILE SCIENCE
Sept 17 (8th Scientific Session, National Research Council, Ottawa, Ontario, Canada)

INTERNATIONAL FEDERATION OF ASSOCIATIONS OF TEXTILE CHEMISTS AND COLOURISTS
Sept 16-18 (London Congress—Church House, Westminster, London, England)

INTER-SOCIETY COLOR COUNCIL
April 11-12, 1960 (29th Annual Meeting—Philadelphia Museum College of Art, Philadelphia, Pa)

NATIONAL COTTON COUNCIL
Oct 7 (Chemical Finishing Conference, Mayflower Hotel, Washington, DC)

SOUTHERN TEXTILE EXPOSITION
Oct 3-7, 1960 (Greenville, SC)

SYNTHETIC ORGANIC CHEMICAL MANUFACTURERS ASSOCIATION OF THE UNITED STATES
Sept 15, Oct 13, Nov 4 (Luncheon meetings at the Hotel Roosevelt, New York, NY); Dec 2 (Annual meeting and dinner—Hotel Roosevelt, New York, NY)

TEXTILE RESEARCH INSTITUTE
March 24-25, 1960 (30th Annual Meeting, Hotel Commodore, New York, NY)

Proceedings of the

American Association of Textile Chemists and Colorists

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September 7, 1959 No. 18

37th National Convention———

THE DEMETALIZATION, LEVELING AND RECHROMING OF CHROME-DYED WOOL PIECES*

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A new method of demetalizing and leveling chrome-dyed pieces is discussed. By the use of Cyquest 40 sequestering agent with certain acids, salts of many metals (including the "tramp" metals) may be removed even after the dye-metal-wool complex has been formed. This new process of demetalization and redyeing has been used practically, and to date, several hundred pieces of fine woolen and worsted fabrics have been leveled by this method and passed as firsts at the perch. The effect of the demetalization method on dyeings made by the top-chrome, meta-chrome and bottom-chrome processes is discussed. A recommended list of the dyes that give excellent results is included.

INTRODUCTION

DYERS of woolen and worsted fabrics have long had the problem of leveling unevenly dyed pieces which have been dyed by a top-, meta- or bottom-chrome procedure. This problem is caused by the fact that few, if any, chrome dyes desorb or migrate after the chromium complex of the dye has been formed within the wool fiber. Consequently, the dyer has had to resort to a stripping and redyeing operation with all its attendent disadvantages, or to redyeing the entire lot into a darker shade. Frequently, the redyed pieces are as uneven as the original pieces, thus necessitating further treatments.

Stripping the material before redyeing may not solve the problem because a) certain of the triarylmethane, anthraquinonoid-, and azochrome dyes are quite resistant to destruction by stripping agents and, therefore, the color may not be removed or destroyed uniformly resulting in a nonlevel bottom; b) the additional operations may chemically damage the wool, thereby increasing its affinity for dye or adversely affecting the hand; and c) the dye that is destroyed is lost, thus resulting in a wasteful and expensive operation.

The presence of iron or other metal ions in woolen and worsted materials usually causes dullness, blotches or unevenness of shade. Unless these metal ions are removed prior to redyeing, the same defects may be produced. The present studies have shown that stripping agents do not remove these metal ions from wool fibers and, therefore, their removal must be achieved by other means. Consequently, there is a need for an improved practical method of leveling which will not damage the wool excessively, which will not require the destruction of dye, and which will remove undesirable ions of metals, such as aluminum, calcium, copper, iron, magnesium and manganese, that might be present.

It has been shown previously by microdyeoscopic studies that certain chromed dyes could be demetalized and returned to their original acid form by the use of a sequesterant.

These studies also showed that, after a metalized dye had been demetalized, desorption and redistribution of the dye throughout the material was possible, thus promoting leveling. Such demetalized and leveled dyeings could then be rechromed in a fresh bath and would, in many cases, produce shades approximately the same as those of the original chromed dyeings. With this background information it seemed desirable to see if a practical solution to this problem could be developed.

During the studies to be reported in this article, a method of leveling by the simultaneous demetalization of the metal-dye complex and removal of metallic impurities was developed which fulfills most of the requirements mentioned above and overcomes the indicated deficiencies.

The method found to give the best results for the greatest number of Calcochrome mordant acid dyes was a two-hour boil in a 40:1 liquor ratio bath containing 8% of oxalic acid, 20% anhydrous sodium sulfate and 2% of Cyquest 40 sequestering agent (tetrasodium ethylenediamine tetra acetate) based on the weight of the material.

The amount of color transferred from the dyed material to the undyed material during the demetalization and leveling treatment gives an indication of the effectiveness of the method. Consequently, each Calcochrome mordant acid dye was studied and the data have been tabulated in column 8 of Table I. These data show that, for most dyes, a comparatively high concentration of dye is transferred. A transfer of 50% of the original dye to the undyed fabric would be perfect because then both samples would contain an equal amount of dye.

Table I also includes the name of each Calcochrome dye, the new *Colour Index* designation and number, the shade of an acid dyeing after it has been subjected to the demetalization and leveling treatment, the shade of the original top-chromed dyeing, the shade of a top-chromed dyeing after it was stripped with sulfoxylate, the percent of dye demetalized by the new process, the strength by reflectance of a demetalized and

*Presented in part before the 37th National Convention at the Hotel Conrad Hilton, Chicago, Ill, on October 30, 1958.

TABLE I

2% Top Chromed Dyeings

(1) Calcochrome Dye	(2) Colour Index Number	(3) Shade of unchromed (acid) dyeing	(4) Shade of unchromed (acid) dyeing after demetalization and leveling treatment	(5) Chromed shade	(6) Shade after stripping with sulfoxylate	(7) Percent dye demetalized by leveling treatment	(8) Percent dye transferred to undyed material during demetalization and leveling treatment (50% is ideal)	(9) Strength by reflectance of demetalized and rechromed dyeing using original top-chromed dyeings as 100%	(10) Percent dye transferred from acid-dyed piece to undyed piece by leveling test (50% is ideal)	(11) Shade change in artificial light (top-chromed dyeing)
Alizarine Blue Black B (CI Mordant Black 13)	63615	dull reddish-violet	redder, brighter	deep reddish-gray	light brown	80	30	50	39	dull black
Alizarine Blue Black 3B (CI Mordant Black 13)	63615	dull bluish-violet	slightly redder	deep greenish-gray	light brown	75	30	85	38	greenish-black
Blue Black R Conc (CI Mordant Black 17)	15705	reddish-violet	bright crimson	navy blue	light greenish-gray	5	20*	50	47	duller, redder
Blue Black ZF Conc (CI Mordant Black 17)	15705	dull maroon	bright crimson	blue black	light greenish-gray	10	20*	80	47	much duller red
Black F Conc (CI Mordant Black 5)	26695	dull navy blue	similar	black	weaker and sl duller	50	7	50	38	duller
Black PV Conc (CI Mordant Black 9)	16500	light reddish violet	much brighter-redder	bluish black	light grayish brown	15	5*	95	31	duller
Black T Ex Conc (CI Mordant Black 11)	14645	brownish-violet	dull maroon	bluish-black	light gray uneven	0	5*	100	37	duller
Brilliant Blue BBG (CI Mordant Blue 1)	43830	reddish-brown	crimson	bright blue	light green	90	50	95	49	much duller, greener
Brown EB (CI Mordant Brown 1)	20110	reddish-orange-brown	brighter yellower	deep bluish brown	light tan sl uneven	50	16	70	31	slightly brighter
Brown EBR (CI Mordant Brown 1)	20110	orange	brighter	deep bluish-brown	light tan	70	15	55	31	brighter, more orange
Brown PG (CI Mordant Brown 40)	17590	brownish-red	duller, yellow-brown	mouse brown	light greenish-gray	40	25	95	47	brighter, more orange
Brown 2R (CI Mordant Brown 13)	13225	yellowish-orange	weaker, duller, browner	dull reddish-brown	light yellower tan	50	15*	50	40	brighter, more
Brown RH (CI Mordant Brown 33)	13250	brownish-orange	brighter, much yellower	dark brown	dull olive, very uneven	85	35	65	43	brighter, more orange
Brown ROC (CI Mordant Brown 33)	13250	orange-red	dull brown	dark brown	dull olive, very uneven	80	20	55	41	brighter, more orange
Orange GR (CI Mordant Orange 6)	26520	bright golden orange	yellower	reddish-orange	very light greenish tan	90	33	70	50	brighter, redder
Orange LC (CI Mordant Orange 8)	—	bright red	dull yellowish orange	orange	dull violet, sl uneven	5	30*	100	49	much brighter
Orange R Ex Conc (CI Mordant Orange 1)	14030	bright, golden yellow	yellower, less red	dull reddish-orange	reddish-yellow, sl uneven	80	47	90	50	brighter, redder
Alizarine Red SC (CI Mordant Red 3)	52005	dull, weak bluish red	dull brownish orange	deep red	dull red-brown, sl uneven	90	43	75	42	brighter, yellower
Fast Red ECB (CI Mordant Red 7)	18760	dull brownish orange	bright reddish orange	bluish red	sl brighter, sl weaker	20	20*	95	45	
Silk White Red B (CI Mordant Red 9)	16105	bright reddish orange	brighter and yellower	medium yellowing red	greenish beige	80	37	100	47	much brighter, yellower
Yellow FC (CI Mordant Yellow 8)	18821	bright yellow	similar	medium yellow	greenish beige	20	37	100	46	much redder
Yellow 2G (CI Mordant Yellow 1)	14025	light yellow	weaker, sl less yellow	deep yellow	mustard	70	50	85	45	much redder
Yellow CGW—250% (CI Mordant Yellow 16)	25100	deep yellow	weaker and brighter	deep reddish yellow	greenish mustard	80	10	90	37	much redder

*Visual estimation.

rechromed dyeing using the original top-chromed dyeing as 100%, the percent of dye transferred from an acid dyeing to an undyed piece by a typical leveling test and finally the shade change in artificial light of an original top-chromed dyeing (columns 1-7 and 9 to 11, respectively).

DISCUSSION AND EXPERIMENTAL

CAUSES OF UNLEVEL DYEING OF CHROME DYES——Although the dyer is usually blamed for unlevel dyeings, experience has proved that the causes of many of the faults found in dyed wool fabrics were present when the goods were received in the dyehouse. The origin of some of these faults often has been traced to physical or chemical damage to the wool which occurred in one or more of the following processes: crabbing, carbonizing, baking,

neutralization, scouring, fulling, chlorination or some other preparatory treatment. Unfortunately, these defects are usually not detected until dye is added to the dyebath, when the faults become apparent through the greater or lesser affinity of the wool for the dye. This type of damage is usually difficult, if not impossible, to cover except in a black.

Another frequent source of unevenness which occurs in applying chromable dyes is the presence of metal ions (salts) in the woolen pieces. These metal ions or metal salts also react with the dye to form metal-dye complexes. Usually the color of these metal-dye complexes is different from the shade of the chrome-dye complex. Slight quantities of contaminating metal ions, when unevenly distributed, can and do cause unevenness and blotches.

Dyes which have strong affinity for wool, ie, milling, neutral-dyeing metalized dyes, and certain chrome dyes, generally have poor leveling properties. These dyes must be exhausted slowly and deposited uniformly on the fabric to obtain a level result. This we have called "exhaustion leveling." Exhaustion leveling is especially important because dyes with poor leveling properties are difficult to remove without drastic treatment once they have penetrated and become fixed within the fiber. Likewise, uneven or shaded pieces dyed by a top-, meta- or bottom-chrome procedure are difficult or impossible to level by the normal methods used for redyeing.

It is well known by dyers that, in general, good leveling is obtained most readily with dyes which exhaust from the bath slowly. Another requirement is that the "color" dye "off-and-on"—in other words, some of the dye must be "desorbed" from the wool. Consequently, the first problem was to study the ease of removing chromed dyes from the colored fabric to see if this approach was practical. This was investigated by means of microscopical studies on wool slubbing which had been dyed separately with Calcochrome Alizarine Red SC (CI Mordant Red 3), Calcochrome Yellow CGW (CI Mordant Yellow 16), and Calcochrome Brilliant Blue BBG (CI Mordant Blue 1) using a top-chrome procedure. (See Appendix, Method No. 2). Equal weights of the red, yellow and blue dyeings and undyed wool were then carded together. These carded blends were boiled for various lengths of time in a fresh bath containing 20% anhydrous sodium sulfate and 5% acetic acid 28%, based on the weight of the wool. Though the dyeings were boiled continuously for two and three hours, the undyed wool remained white, or only faintly tinted, and little, if any, change could be noted in the red, yellow and blue shades, indicating that desorption did not occur.

When the above experiment was repeated using easily leveling acid dyes, the dyeings were all colored the same brown shade, indicating that desorption and redistribution of dye did occur.

The above results were further confirmed by using top-chromed dyeings on wool pieces of Calcochromes Blue Black R (CI Mordant Black 17), Silk White Red B (CI Mordant Red 9), Yellow FC (CI Mordant Yellow 8), Alizarine Red SC (CI Mordant Red 3), and Brown RH (CI Mordant Brown 33). The particular purpose of this group of experiments was to determine the amount and condition of the dye, if any, which was transferred from the dyed to the undyed wool. In these experiments, the undyed wool was tinted or only lightly colored in all cases. Subsequent chroming of the tinted wool failed to develop more than a slight increase in color value. These experiments indicated that most of the desorbed dye was metalized. They also indicated that the problem of leveling could not be solved by the desorption of the metal-dye complex.

LEVELING OF UNMETALIZED CHROMABLE DYES——The next step was to study the leveling characteristics of the unmetalized chromable dyes in their acid form (self-shades). It was important to know whether these unmetalized dyes generally behaved like the acid dyes referred to above or whether they behaved like the milling or the top-chromed dyes, in so far as desorption was concerned.

It was found that chrome dyes varied considerably in their leveling properties before chromation and ranged from the crystalloidal acid-insensitive dyes, which leveled without difficulty, to colloidal dyes, which are extremely acid sensitive and level only with difficulty. The crystalloidal dyes leveled well under normal dyeing conditions, some leveling perfectly when added to a boiling sulfuric acid bath, eg, CI Mordant Yellow 8. On the other hand, many of the colloidal dyes had leveling properties quite similar to those of the milling dyes and varied from unlevel to moderately level dyeing types, eg, CI Mordant Black 5.

In order to determine how well mordant acid dyes desorb and level in the acid state, a series of experiments was made in which each dye was applied by Method No. 1, and then was subjected to a leveling test by boiling a five-gram dyeing (unchromed) with a five-gram piece of undyed wool flannel for one hour in a bath containing 5% acetic acid 28% (owf) and 20% anhydrous sodium sulfate using a bath ratio of 40:1 and a starting pH of 5.0. The percent of dye transferred from the piece dyed with the unchromed dye to the undyed piece was measured spectrophotometrically and the results are tabulated in Column 10 of Table I. These data indicate that the unchromed color dyes "off-and-on," ie, that it desorbs. The data also indicate that a satisfactory leveling condition might be brought about if it were possible to cleave the wool-dye-chrome complex and return the dye to its original unchromed state. When it was found that most mordant acid dyes desorbed and leveled so well in their acid form, it was believed that, if the wool-dye-chrome complex could be broken and the chrome removed without detrimental effect on the wool or the dye, it might also be possible to remove other metallic-ion contaminants at the same time. Such a process, if practical, would certainly be superior to stripping. It would also avoid the waste of the dye originally used and should make level dyeing much easier during the redyeing step.

SEQUESTERING AGENTS——It was decided to begin this investigation by studying the effects of sequestering agents, either alone or in combination with a compound such as oxalic acid on chrome-dyed wool piece goods. Oxalic acid was of special interest because of its known property of removing iron stains from textiles, and because earlier unpublished Microdyeoscopic studies in this laboratory had shown that the wool-dye-chrome complex of Calcochrome Brilliant Blue BBG (CI Mordant Blue 1) and Calcochrome Cyanine RC could be completely demetalized and returned to their unchromed shades by treatment in a normal oxalic acid solution for 30 to 60 minutes at 212°F. When these demetalized dyeings were again top-chromed (rechromed), they returned to the original top-chromed shade except for a few dyes where some of the original color was lost.

A search of the literature produced a few references on the use of demetalizing agents in redyeing. Eastwood (*1*) mentioned that in redyeing

he found a good boil with oxalic acid and a strip with ammonia removed sufficient color for his purpose. Townend (2) stated that reduction is facilitated by preboiling the material in citric or oxalic acids. The Philadelphia Section of the AATCC, in an Intersectional Contest paper (3), described an experiment using oxalic acid. Heuthwaite (4) has stated that, when shades are too dark, it is necessary to treat the goods in a warm bath with anhydrous sodium sulfate and oxalic or sulfuric acids in order to partially split the color lake. Bird (5) states that chrome dyes can sometimes be largely removed from wool by just splitting the dye lake by means of a treatment with a 0.5-1.0% solution of a mixture of hydrochloric and oxalic acids for 20 minutes at a temperature just under the boil, and then treating with a hot dilute ammonia solution. None of these references, except Bird, contained any information as to the amounts of chemicals used, the temperature, the duration of treatment or the specific results obtained.

Since new and efficient sequestering agents have been developed in recent years, these agents and a combination of these agents with oxalic acid were also evaluated.

Of all the methods of applying chromable dyes to wool, the top-chrome method is the most sensitive to shade change by undesirable trace metal ions. Since the dye is applied in the first step and chromed in a second step, the presence of any trace metals, such as iron, aluminum, calcium, copper, magnesium or manganese, affords an opportunity for these metals to react with the dye and form metal complexes prior to top chroming. These dye-metal complexes may be colored differently from the chrome-dye complex and may show up as blotches.

The procedure for top-chrome dyeing is described in Method No. 2 in the Appendix. Wool flannel pieces were used. After the dyeings were completed, the dyed pieces were rinsed and then entered into the demetalization bath.

After trying many different demetalization baths consisting of oxalic acid alone or in combination with various sequestering agents, we found that Method No. 6, as stated previously, gave the best results with the most dyes and appeared to be the most practical.

This method uses a combination of 2% Cyquest 40 sequestering agent, 8-10% oxalic acid and 20% sodium sulfate at a liquor ratio of 30:1. With certain mordant acid dyes, common salt produces better results than anhydrous sodium sulfate, insofar as demetalization is concerned, but the comparative leveling properties under these conditions were not investigated.

TABLE II

Demetalization of Calcochrome mordant acid dyes applied by the top-chrome method and demetalized with 8% oxalic acid, 2% Cyquest 40, and 20% anhydrous sodium sulfate for two hours at the boil

Group I—Completely and Easily Demetalized	
Calcochrome	Alizarine Blue Black B (CI Mordant Black 13)
"	Alizarine Blue Black 3B (CI Mordant Black 13)
"	Brilliant Blue BBG (CI Mordant Blue 1)
"	Brown RH (CI Mordant Brown 33)
"	Brown ROC (CI Mordant Brown 33)
"	Orange GR (CI Mordant Orange 6)
"	Orange R Ex Conc (CI Mordant Orange 1)
"	Alizarine Red SC (CI Mordant Red 3)
"	Silk White Red B (CI Mordant Red 9)
"	Yellow 2G (CI Mordant Yellow 1)
"	Yellow CGW 250% (CI Mordant Yellow 16)
"	Cyanine RC
Group II—Moderately Demetalized	
Calcochrome	Blue Black R (CI Mordant Black 17)
"	Black F (CI Mordant Black 5)
"	Brown EB (CI Mordant Brown 1)
"	Brown EBR (CI Mordant Brown 1)
"	Brown PG (CI Mordant Brown 40)
"	Brown 2R (CI Mordant Brown 13)
"	Fast Red ECB (CI Mordant Red 7)
Group III—Slightly Demetalized	
Calcochrome	Blue Black ZF (CI Mordant Black 17)
"	Black T Conc (CI Mordant Black 11)
"	Yellow FC (CI Mordant Yellow 8)
"	Black PV Conc (CI Mordant Black 9)
Group IV—Not Demetalized	
Calcochrome	Orange LC (CI Mordant Orange 8)

Dyeings were then made with the line of Calcochrome mordant acid dyes using the top-chrome method (Method No. 2) after which they were demetalized using Method No. 6, and then rechromed. The results are given in Table II, in which the dyes are listed in four groupings depending upon their ease of demetalization.

To illustrate the effectiveness of the treatment, the following families of spectrophotometric curves for several Calcochrome mordant acid dyes were made and are shown in Figures 1, 2, 3, 4 and 5. With the exception of Calcochrome Alizarine Red SC (CI Mordant Red 3) (Figure 4), the top chromed and top chromed, demetalized and rechromed dyeing curves of the same dye are quite similar. These curves show that in most cases this demetalization and leveling treatment does not destroy or remove the dye.

DEMETALIZATION OF META-CHROME DYEINGS BY METHOD NO. 6———Two percent dyeings of Calcochrome Yellow 2G (CI Mordant Yellow 1), Orange GR (CI Mordant Orange 6), Cyanine RC, Brilliant Blue BBG (CI Mordant Blue 1), Alizarine Red SC (CI Mordant Red 3), Brown RH (CI Mordant Brown 33), and Violet B were made by Method No. 3. (See Appendix). These dyeings were then demetalized by the procedure outlined in Method No. 6. All dyeings were completely demetalized, or demetalized to a greater extent than top-chrome dyeings made with the same dye. Most of the meta-chrome dyeings when boiled one hour were demetalized to approximately the same extent as those boiled for two hours.

The results of the above experiments indicate that meta-chrome dyeings are demetalized more easily or to a greater extent than top-chrome dyeings.

DEMETALIZATION OF BOTTOM-CHROME DYEINGS BY METHOD NO. 6———Two percent dyeings of Calcochrome Yellow 2G (CI Mordant Yellow 1), Brilliant Blue BBG (CI Mordant Blue 1), Violet B, Blue Black R Conc (CI Mordant Black 17), and Alizarine Blue Black B (CI Mordant Black 13) were made by Method No. 4. These dyeings were then demetalized by the procedure outlined in Method No. 6. The Yellow 2G, Blue BBG and Alizarine Blue Black B were completely demetalized while the Blue Black R and Violet B dyeings were demetalized to a much greater extent than the top-chrome dyeings.

These preliminary meta- and bottom-chrome studies indicate that the type of wool-dye-chromium complex formed by these methods is more readily demetalized than the complex formed when the same dyes are applied to wool by a top-chrome procedure.

REMOVAL OF CONTAMINANTS BY METHOD NO. 6———Metal salts such as iron, aluminum, copper manganese, magnesium and calcium are often present as contaminants in dyes, chemicals, and the water used in dyehouse operations. Many of these metal salts, and especially those of iron, are present as natural impurities in water or may be picked up when the water passes through rusty iron pipes. Such salts, especially those of iron, have an affinity for wool and will exhaust almost completely from an acid dye-

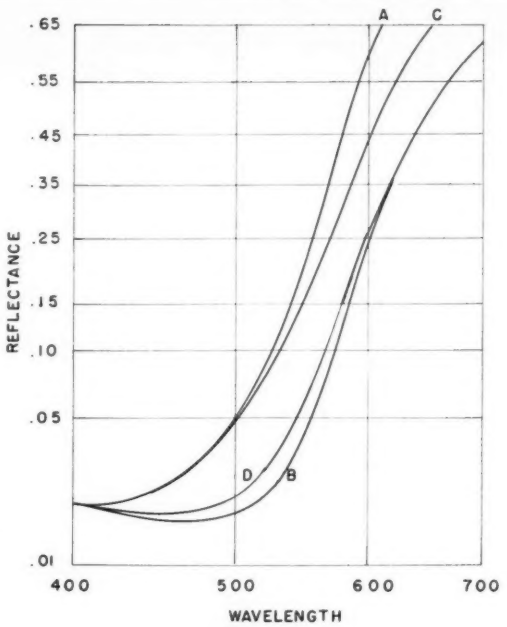

Figure 1

2% Calcochrome Orange GR (CI Mordant Orange 6) CI 26520

Curve A———Acid dyeing, Method No. 1.
Curve B———Top-chromed dyeing, Method No. 2.
Curve C———Top-chromed, demetalized dyeing, Method No. 2 followed by Method No. 6.
Curve D———Top-chromed, demetalized and rechromed dyeing, Method No. 2 followed by Method No. 6 followed by Method No. 7.

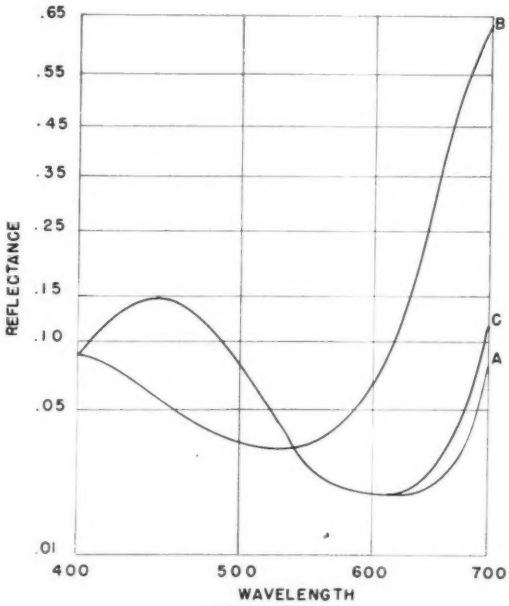

Figure 3

2% Calcochrome Brilliant Blue BBG (CI Mordant Blue 1) CI 43830

Curve A———Top-chromed dyeing, Method No. 2.
Curve B———Demetalized top-chromed dyeing, Method No. 2 followed by Method No. 6.
Curve C———Top-chromed dyeing, demetalized and rechromed, Method No. 2 followed by Method No. 6 followed by Method No. 7.

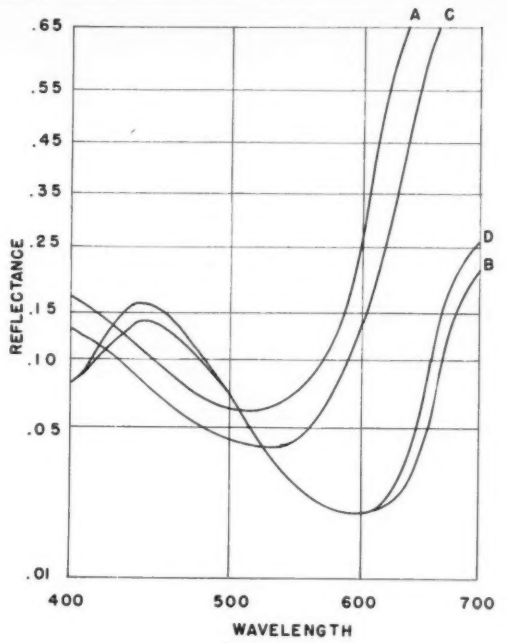

Figure 2

2% Calcochrome Cyanine RC

Curve A———Acid dyeing demetalized, Method No. 1 followed by Method No. 6.
Curve B———Top-chromed dyeing, Method No. 2.
Curve C———Demetalized top-chromed dyeing, Method No. 2 followed by Method No. 6.
Curve D———Top-chromed dyeing, demetalized, and rechromed, Method No. 2 followed by Method No. 6 followed by Method No. 7.

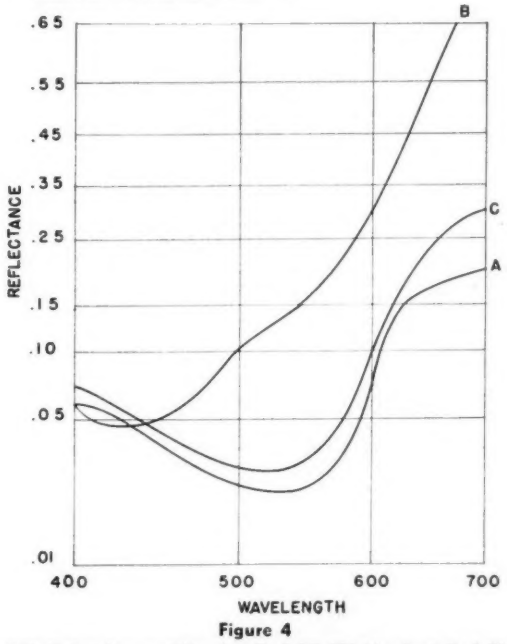

Figure 4

2% Calcochrome Alizarine Red SC (CI Mordant Red 3) CI 52005

Curve A———Top-chromed dyeing, Method No. 2.
Curve B———Demetalized top-chromed dyeing, Method No. 2 followed by Method No. 6.
Curve C———Top-chromed dyeing, demetalized and rechromed, Method No. 2 followed by Method No. 6 followed by Method No. 7.

bath. Even so, it is often difficult to prove that metal salts are responsible for unevenness and blotches in dark shades.

A large number of experiments was run to determine the ease of demetalization of the iron, copper and aluminum complexes of chromable dyes, Figures 6, 7 and 8. If this could be accomplished, a procedure of practical importance would be available for the dyer. The requirement here would be for the undesired metal-dye complex to demetalize more readily than the desired chromium complex. Such a demetalization treatment would remove the metal and level the pieces without complete demetalization of the chromium complex. This would be especially useful when dyeing with Calcochrome Black T Conc (CI Mordant Black 11), Orange LC (CI Mordant Orange 8) and Black PV Conc (CI Mordant Black 9), whose chromium complexes are quite resistant to demetalization by any of the methods tried. In all cases, dye complexes of these dyes with iron, copper and aluminum were demetalized, either more readily or as readily as the chromium complex.

These studies were then extended to include other metal complexes. Experiments have shown that Calcochrome Alizarine Red SC (CI Mordant Red 3), Orange GR (CI Mordant Orange 6) and Black PV (CI Mordant Black 9) topped with cobalt, nickel, silver, bismuth and molybdenum in most cases produced metal complexes whose shades were unlike those of the unmetalized or chromed shades. These dyeings were then subjected to the demetalization treatment, and in all cases with the exception of the dyeing stopped with silver and the cobalt shade of Black PV, they were easily demetalized.

DIFFUSION OF OXALIC ACID ———Since, as stated previously, oxalic acid has been used in demetalization leveling baths, it seemed desirable to evaluate some of the properties of this acid and especially the rate of its diffusion into the dyed fiber. It was found that the rate of diffusion of oxalic acid into wool fibers is extremely rapid. This was determined by a microscopical study of cross sections made from 3% dyeings of Calcochrome Brilliant Blue BBG (CI Mordant Blue 1) demetalized with 1 to 8% oxalic acid. If the rate of diffusion of oxalic was slow, the fibers would show an outer ring or the demetalized-dye shade with a core having the shade of the original chromed dye which is blue. As indicated in Table III, for all concentrations of oxalic acid used, demetalization appeared to take place evenly throughout the fiber.

TABLE III

One hour boiling oxalic acid treatment of worsted yarn which had been dyed top-chrome by Method No. 2 with 3% Calcochrome Brilliant Blue BBG (CI Mordant Blue 1)

Oxalic acid (%)	*Penetration*	*Color of fiber*
1	complete	bright blue
2	complete	bluish-violet
3	complete	violet
4	complete	light violet
5	complete	dark red
6	complete	red
7	complete	red
8	complete	light red

CHANGES IN SHADE CAUSED BY DEMETALIZATION———The demetalization treatment often causes extreme changes between the shade of the unmetalized acid dyeing and the shade of the same dyeing after top chroming and demetalization. These extreme color changes interfered with the spectrophotometric method developed by Stearns (private communication) for the determination of the percent of dye demetalized. In the Stearns' method, the unchromed acid shade of the dye is used as a standard (100% unmetalized), and the top-chromed dyeing is used as the 100% metalized standard. For example, the unchromed dyeing of Calcochrome Alizarine Red SC (CI Mordant Red 3) is a dull, weak, bluish-red. When this dye is applied by a top-chrome or metachrome procedure and then demetalized, the shade is a dull brownish-orange. Similarly, Calcochrome Brilliant Blue BBG (CI Mordant Blue 1) has a reddish-brown shade when it is unchromed, and a bright crimson shade when a top-chromed dyeing has been subsequently demetalized.

These shade changes were investigated and it was established that they were due mainly to differences in pH, and to a lesser degree, to the sequestering of trace metals such as iron, copper, aluminum and other metals which form complexes with chromable dyes and whose shades are different from those of the chromium-dye complex.

In order to increase the accuracy of the spectrophotometric determinations, unmetalized acid dyeings of all Calcochrome mordant acid dyes were subjected to the demetalization and leveling (Method No. 6). The original and the demetalized dyeings were then compared visually, and the differences in shade are described in Table I, Columns 3 and 4. Spectrophotometric curves showing these differences are in Figures 9, 10 and 11. With some dyes, these differences are so great that these data may prove to be of value for reference purposes to dyers who may use the demetalization process for leveling.

BOILING TIME REQUIRED FOR DEMETALIZATION———From this study, the time required for demetalization in Method No. 6 was developed. This method includes a two-hour period at the boil to insure as complete demetalization as is possible. In developing this method, a sample was removed from each dyeing after boiling in the demetalization bath for one hour and it was observed that many dyeings were completely demetalized after one hour at the boil. Others, though not completely demetalized, changed very little between the one- and two-hour periods of boiling. A number of dyeings, however, required a full two-hour boil to obtain the maximum degree of demetalization. If the dyer should be confronted with a problem of leveling, samples of each shade to be leveled should be checked first in the laboratory to establish the time and the percent of sequesterants required to produce maximum demetalization within a minimum of time. However, it is not always necessary or desirable to have complete demetalization of the chromium complex.

INFLUENCE OF LIQUOR RATIO ———It has been shown by laboratory experiments and also by practical runs that the liquor ratio of the demetalization bath influences the rate and extent of demetalization. To obtain the maximum effect of the chemicals, it is necessary to use the lowest liquor ratio possible. The ratio recommended as a result of this study is no higher than 30:1 (see Method No. 6). When the liquor ratio is less than 30:1, the percentage of each chemical may be reduced. With longer liquor ratios, the percentage of each chemical should be increased, the exact percentages, of course, depending upon the ease with which the specific dye or combination of dyes is demetalized.

A STUDY OF DEMETALIZING FORMULAS———Calcochrome Black PV (CI Mordant Black 9) was selected as the dye to use in the evaluation of different chemicals and combinations of chemicals because it

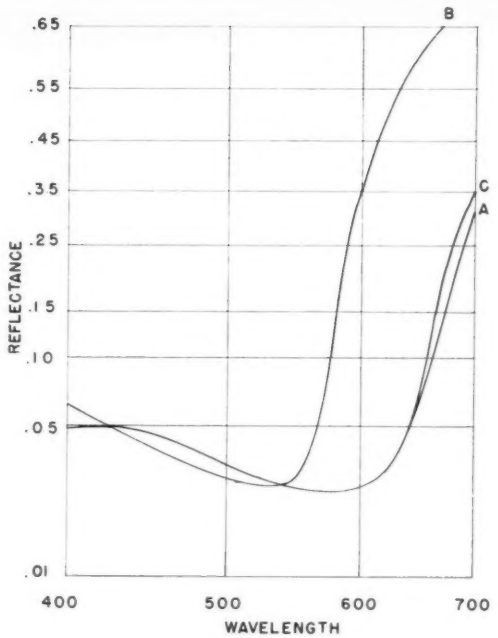

Figure 5

2% Calcochrome Violet B
Curve A————Top-chromed dyeing, Method No. 2.
Curve B————Demetalized top-chromed dyeing, Method No. 2 followed by Method No. 6.
Curve C————Top-chromed dyeing demetalized and rechromed, Method No. 2 followed by Method No. 6 followed by Method No. 7.

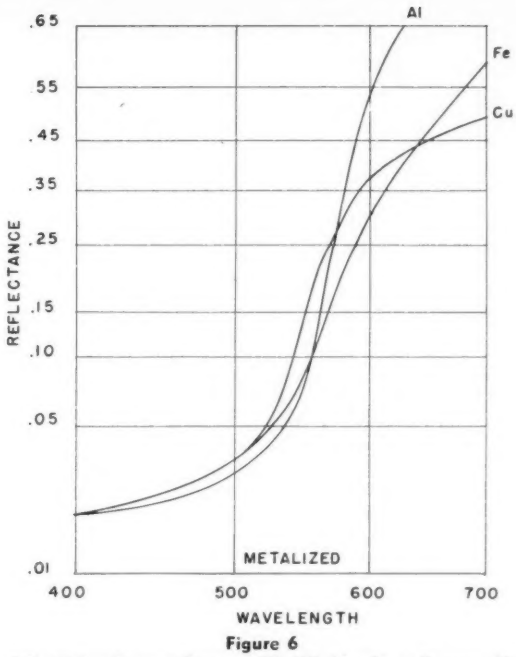

Figure 6

2% Calcochrome Orange GR (CI Mordant Orange 6) CI 26520
Curve Al—Acid dyeing topped with 2% aluminum sulfate, $Al_2(SO_4)_3$.
Curve Fe————Acid dyeing topped with 1% ferrous sulfate, $Fe\ SO_4$.
Curve Cu————Acid dyeing topped with 1% cupric sulfate, $Cu\ SO_4$.

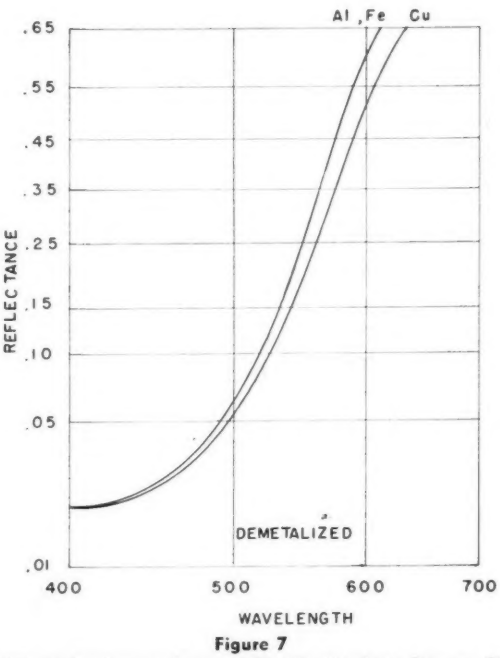

Figure 7

2% Calcochrome Orange GR (CI Mordant Orange 6) CI 26520
Curves Al, Fe and Cu————Metalized dyeings demetalized by Method No. 6.

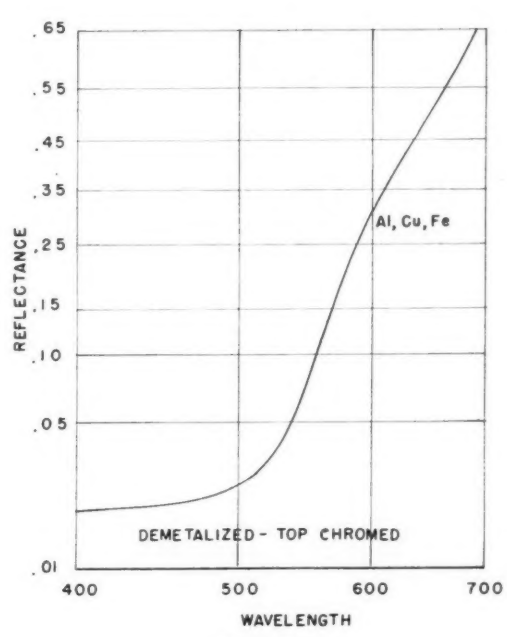

Figure 8

2% Calcochrome Orange GR (CI Mordant Orange 6) CI 26520
Curve Al, Cu, Fe————Metalized dyeings, demetalized and rechromed, Method No. 6 followed by Method No. 7.

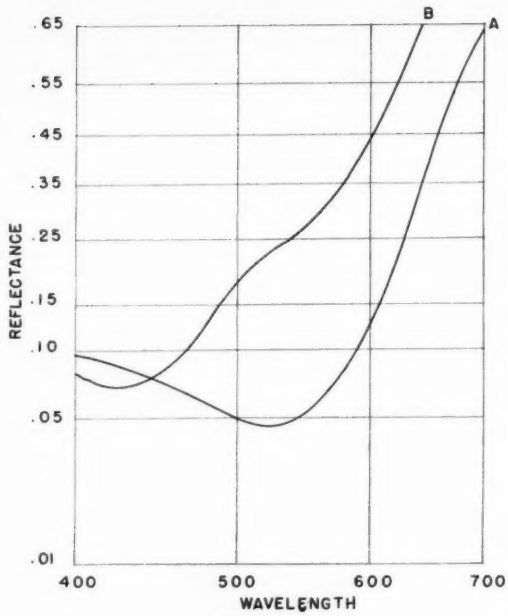

Figure 9

2% Calcochrome Alizarine Red SC (CI Mordant Red 3) CI 52005
Curve A———Acid dyeing, Method No. 1.
Curve B———Acid dyeing and demetalized, Method No. 1 followed by Method No. 6.

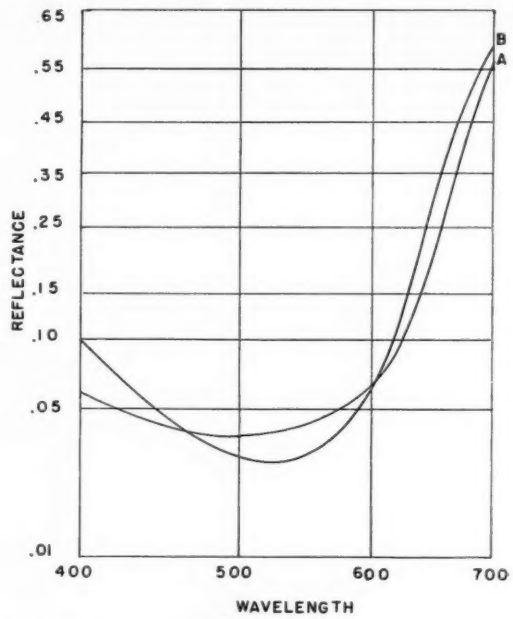

Figure 10

2% Calcochrome Brilliant Blue BBG (CI Mordant Blue 1) CI 43830
Curve A—Acid dyeing, Method No. 1.
Curve B———Acid dyeing and demetalized, Method No. 1 followed by Method No. 6.

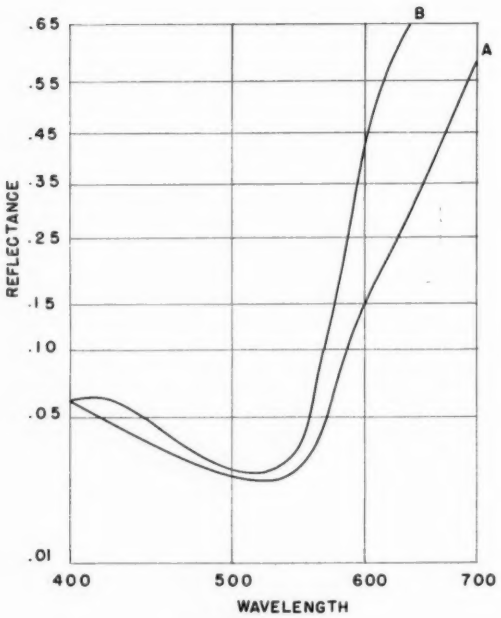

Figure 11

2% Calcochrome Violet B
Curve A———Acid dyeing, Method No. 1.
Curve B———Acid dyeing demetalized, Method No. 1 followed by Method No. 6.

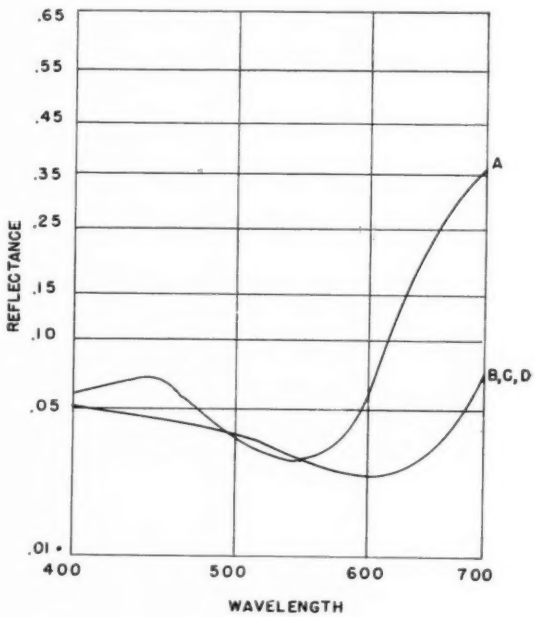

Figure 12

2% Calcochrome Black PV Conc (CI Mordant Black 9) CI 16500
Curve A———Acid and demetalized acid dyeing, Method No. 1 followed by Method No. 6.
Curve B, C, D
B, top-chromed
C, top-chromed and demetalized
D, top-chromed, demetalized and rechromed.

TABLE IV

Sequestering Agents

Mono-, di-, and trisodium salts of ethylenediamine tetra-acetic acid and the iron specific type.

Cyquest 40 Sequestering Agent

Acids	*Salts*
oxalic acid	ammonium chloride
citric acid	ammonium sulfate
tartaric acid	ammonium acetate
phytic acid	ammonium thiocyanate
aconitic	barium chlorate
acetic	diethyl oxalate
formic	hydroxyl ammonium sulfate
latic	magnesium sulfate
nitric	potassium chlorate
phosphoric	potassium sulfite
sulfuric	sodium bisulfite
	sodium chloride
	sodium chlorate
	sodium nitrite
	sodium sulfate
	trisodium phosphate

Organic compounds	*Surface-active agents*
cerelose	Calcofast salt
pyridine	Calcomet salt
	Deceresol NY

is only slightly demetalized by Method No. 6. This dye was also a good choice because its acid (unmetalized) shade is a reddish-violet while its chromed shade is a black which makes the shades easy to distinguish. These differences are shown by spectrophotometric curves in Figure 12.

A large number of compounds, and combinations of these compounds, were tried using the degree of demetalization produced by Method No. 6 as the standard for comparison purposes. The chemicals examined are listed in Table IV.

Increasing the percentage of oxalic acid from 10%, to 25%, 50%, 75% and 100% gave improved results up to 50%. Beyond this amount, the improvement was slight. Substituting equivalent amounts of citric or tartaric acids for oxalic acid did not produce results equal to those obtained with oxalic. Phytic acid had little, if any, effect upon demetalization. Several combinations gave slightly to definitely superior demetalization. (See Table V.) The substitution of common salt for sodium sulfate gave a greater degree of demetalization. However, the comparative leveling properties of sodium chloride versus sodium sulfate in the demetalization process would have to be determined since leveling is also important. This has not been done.

Table V shows the combinations which gave a greater degree of demetalization than is obtained by Method No. 6.

Experiments have shown that dyeings topped with metals can be demetalized with oxalic acid only, but subsequent chromation produces a duller shade than the standard top-chromed dyeing. The reason for this

TABLE V

	Oxalic Acid (%)	*Sodium Sulfate* (%)	*Cyquest 40* (%)	*Common salt* (%)
Method No. 6	10	20	2	—
Much better	20	20	5	—
Better	10	—	5	20
Somewhat better	10	20	5	5 sulfuric acid
Somewhat better	10	20	—	10 citric acid
Somewhat better	10	20	—	10 tartaric acid
Somewhat better	10	20	5	10 hydroxyl ammonium sulfate

difference was investigated and it was found that demetalization by oxalic acid breaks the chelate bond, but does not remove the metal from the wool. Apparently the metal combines with the wool and is tightly bound though it is capable of metalizing the dye. When oxalic acid and Cyquest 40 sequestering agent are used in combination, the oxalic acid breaks the dye-metal complex and the Cyquest 40 removes most of the metal from the wool fiber. Subsequent rechroming then produces much brighter shades.

A series of experiments was run using 2% Calcochrome Violet B topped with 1% ferrous sulfate. Demetalization with 10% oxalic acid (owf) returned the shade to that of the acid dyeing. However, when the dyeings were rechromed, the shade was duller than a top-chromed dyeing, which indicated the presence of iron dye complex. This shows that all of the iron was not removed by the oxalic acid.

EFFECT OF CONCENTRATION OF BICHROMATE ON DEMETALIZATION——The effect of the concentration of chrome used for top-chroming dyeings was studied to determine its effect upon subsequent demetalization. Two percent dyeings of Calcochrome Black PV Conc (CI Mordant Black 9) and of Blue Black R Conc (CI Mordant Black 17) were topped with 0.25%, 0.5%, 0.75%, 1.0%, 1.5% and 2.0% sodium bichromate, respectively. (Method No. 2). The difference in shade between the dyeings chromed with 0.25% and 2.0% was not very great. All dyeings were subjected to Method No. 6, each in a separate bath.

The shades of the demetalized dyeings which had been top-chromed with 0.25% sodium bichromate were much closer to the unmetalized shade than any of the others, possibly due to incomplete chromation of the original acid dyeing. As the percentage of bichromate was increased, the extent of demetalization decreased. These tests indicate that the less chrome used, the greater the extent of demetalization.

REMOVAL OF ALUMINUM, COPPER AND IRON SPOTS AND BLOTCHES —— Calcochrome Orange LC (CI Mordant Orange 8) cannot be demetalized by Method No. 6. It was, therefore, important to know if the iron and copper complexes of this dye could be demetalized in the presence of the wool-chrome-dye complex. A dyeing was topped with iron, and a second dyeing was topped with chrome. Both dyeings were then treated in the same bath using Method No. 6. A similar set was run using a dyeing topped with copper along with a chrome-topped dyeing. The treatment completely demetalized the iron and copperized dyeings and returned then to the shade of the unmetalized dye, while the top-chromed dyeings showed little, if any, change in shade.

Tramp metals and especially iron often cause staining, blotches and unevenness in chrome-dyed pieces. In order to study the elimination of these stains and unevenness, pieces of undyed wool flannel were spotted with hot one percent solutions of ferric chloride, ferrous sulfate, cupric sulfate and aluminum sulfate and allowed to air dry. These pieces were then dyed with Calcochrome Fast Red ECB (CI Mordant Red 7), Alizarine Red SC (CI Mordant Red 3), and Blue Black R Conc (CI Mordant Black 17), respectively; all these dyes having chromium complex shades distinctly different from the shades produced with aluminum, copper and iron.

All of the spotted areas were easily located by a difference in shade as well as by the presence of a knotted thread of cotton which had been fastened to the center of the spot. These dyeings were then demetalized by the standard Method No. 6. Examination of these dyeings showed that all were perfectly level, uniform in shade, and it was not possible to detect the spots. These experiments showed that unevenness and blotches could be removed completely by the demetalization treatments as shown in Figures 13, 14, 15 and 16.

In another experiment, five-yard pieces of wool flannel were spotted

Figure 13

Wool fabric spotted with ferrous sulfate, cupric sulfate and aluminum sulfate solutions, dried and then dyed.
2% Calcochrome Alizarine Red SC (CI Mordant Red 3) CI 58005
8% Acetic acid 28%
Liquor ratio 30:1
Boiled 1.5 hours.

Figure 14

Spotted and dyed as in Figure 13 then top-chromed in the dyebath with 1.0% sodium bichromate.
Boiled 45 minutes.

with one percent solutions of aluminum sulfate, cupric sulfate and ferrous sulfate, respectively, and allowed to air dry for 12 hours. The pieces were then dyed with Calcochrome Orange R (CI Mordant Orange 1), demetalized by Method No. 6 and top-chromed to simulate a large-scale plant operation. All spots were removed and the pieces were perfectly level.

A series of four dyeings was made by staining a large area of wool flannel with a hot solution of ferrous sulfate, allowing to dry overnight, and all were dyed with 2% Calcochrome Orange LC (CI Mordant Orange 8). All pieces showed the presence of iron by being brown in shade in the spotted area, ie, the iron complex of Calcochrome Orange LC (Figure 17). Three pieces were then top-chromed and the spotted area was dark brown (Figure 18). Two pieces of the top-chromed dyeings were then demetalized by Method No. 6 and the spots disappeared (Figure 19). One piece was then rechromed and was uniform in shade and free from blotches (Figure 20).

These tests are of practical interest because unevenness or blotches in woolen and worsted pieces caused by metalization of the dye by tramp metals, and especially those of iron, can be remedied by the demetalizing and leveling treatment even though the top-chromed dye cannot be demetalized.

CHEMICAL ANALYSIS OF DYED AND UNDYED WOOL FOR CHROMIUM——Chemical analyses for chromium were made on undyed wool which had been subjected to "mock dyeing" processes without dye. One piece was treated by a top-chrome procedure, another piece was top-chromed and then demetalized, a third piece was top-chromed, demetalized and top-chromed a second time in the absence of dye.

A duplicate series was run using 2% Calcochrome Orange GR (CI Mordant Orange 6) on each dyeing. These dyeings were then analyzed by microchemical methods for chromium content. The results of both sets of analyses are listed in Table VI.

RECHROMING DEMETALIZED DYEINGS —— The chromium which remains in the wool after demetalization has been shown by experiments to be available for the rechromation of metalizable dyes and, therefore, the amount of bichromate to be added for rechroming can be reduced considerably. A series of

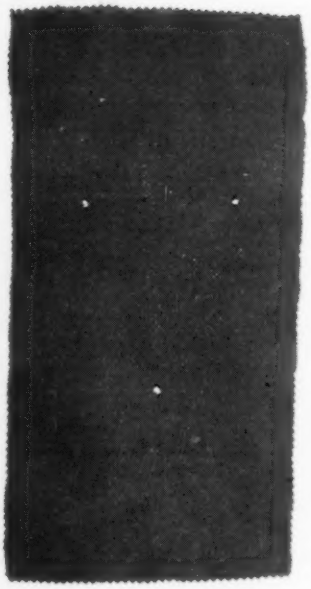

Figure 15

Spotted, dyed and top-chromed as in Figures 13 and 14, then demetalized and leveled with 2.0% Cyquest 40, 8.0% oxalic acid and 20% anhydrous sodium sulfate. Boiled 1.5 hours.

Figure 16

Spotted, dyed, top-chromed, demetalized and leveled as in Figures 13, 14, and 15. Then rechromed in a fresh bath with 1.0% sodium bichromate and 4.0% acetic acid 28%. Boiled 45 minutes.

Figure 17
Wool fabric spotted with ferrous sulfate solution, dried and then dyed.
2% Calcochrome Orange LC (CI Mordant Orange 8)
8% Acetic acid 28%
Liquor ratio 30:1
Boiled 1.5 hours.

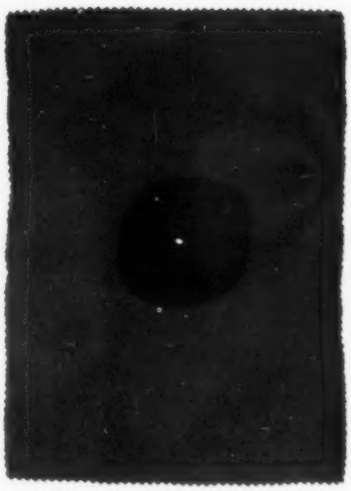

Figure 18
Spotted and dyed as in Figure 17 then top-chromed in the dyebath with 1.0% sodium bichromate. Boiled 45 minutes.

2% dyeings of Calcochrome mordant acid dyes which were top-chromed by Method No. 2 was demetalized and then rechromed with 0.6% of bichromate in place of the usual 1%. The 0.6% bichromate dyeings were equal in all cases to the original shades obtained with 1% chrome, and in several cases, notably Fast Red ECB (CI Mordant Red 7), Black F (CI Mordant Black 5), and Alizarine

TABLE VI

	Chromium on weight of wool (%)
Undyed wool—top-chrome procedure	0.113
Undyed wool—top-chromed and demetalized	0.086
Undyed wool—top-chromed, demetalized and rechromed	0.248
2% Calcochrome Orange GR—top-chromed	0.130
2% Calcochrome Orange GR—top-chromed and demetalized	0.047
2% Calcochrome Orange GR—top-chromed, demetalized and rechromed	0.147

Blue Black B (CI Mordant Black 13), the lesser amount of bichromate produced brighter and deeper dyeings.

Experiments have shown that the demetalized dyeings of certain chrome dyes will not produce the original shade when rechromed by Method No. 7. Calcochrome Fast Red ECB is much weaker, Blue Black R is weaker and greener in shade, and Black F is a yellowish-brown instead of a black.

These radical shade changes have been investigated using Calcochrome Black F as an example. Various modifications of the rechroming operation after demetalization were tried. The following procedure gave the best results: 2.0% chrome alum, 1.0% ammonium hydroxide and 5.0% ammonium acetate. The dyeing was chromed for 30 minutes at the boil. When a demetalized dyeing of Black F is treated as above, the shade is a bluish-black rather than a yellowish-brown.

EFFECT OF DEMETALIZATION TREATMENT ON TENSILE STRENGTH———Tensile strength tests were made on wool flannel which had been dyed by the standard top-chrome Method No. 2, and on similar top-chromed dyeings which had been demetalized, followed by rinsing, and then rechromed in a fresh bath as outlined in Method No. 7. These tests indicated that the demetalization treatment did not cause excessive tendering since only minor losses occurred. However, some variation may occur depending upon the past history of the wool, ie, fleece, chlorinated or stripped. The construction of the fabric also exerts an influence.

EFFECT OF DEMETALIZATION AND RECHROMING ON FASTNESS——Some demetalized and rechromed dyeings are deeper in shade than the original dyeing. Other dyeings become weaker in shade and

Figure 19
Spotted dyed, top-chromed as in Figures 17 and 18. Then demetalized and leveled with 2.0% Cyquest 40, 8.0% oxalic acid and 20% anhydrous sodium sulfate. Boiled 1.5 hours.

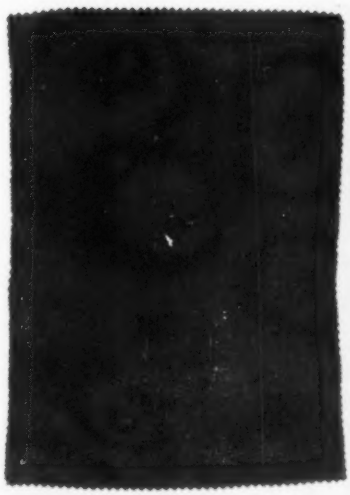

Figure 20
Spotted, dyed, top-chromed, demetalized and leveled as in Figures 17, 18, and 19. Then rechromed in a fresh bath with 1.0% sodium bichromate and 4.0% acetic acid 28%. Boiled 45 minutes.

require an addition of one or more dyes in order to match the standard. Many chrome dyes can be added to the demetalization bath so that demetalization and redyeing proceed simultaneously. Dyes used for such purposes must be acid-insensitive types which remain in solution and level well at a low pH.

For these tests, top-chromed dyeings were made by Method No. 2,

and these dyeings were then demetalized, leveled and redyed simultaneously. After being rechromed in a fresh bath, the dyeings were rinsed thoroughly and dried.

The original and redyed dyeings were then subjected to AATCC Fulling Test No. 3, and exposed in the Fade-Ometer for 40 to 80 hours. A comparison between the two sets of tests showed very little difference in fastness properties.

DEMETALIZATION OF CHROME-DYED NYLON —— Exploratory dyeings using twelve Calcochrome mordant acid dyes applied to nylon by the top-chrome procedure and then demetalized showed that nylon fabrics would produce approximately the same results as wool and would permit the use of this procedure on wool-nylon blends.

PRACTICAL APPLICATION———The demetalization and leveling treatment has been used practically, and to date several hundred uneven pieces of fine woolens and worsted fabrics have been leveled by this method and passed as firsts at the examining perch. No complaints on these pieces have been received from the trade. Several large lots of unevenly dyed top-chromed wool raw stock have also been demetalized and leveled by this procedure.

SUMMARY

A practical procedure has been developed for demetalizing and leveling the dye in fabrics unevenly dyed by the conventional top-, meta- and bottom-chrome methods, comprising treating the unevenly dyed fabrics with demetalizing baths containing Cyquest 40 sequestering agent, oxalic acid, and anhydrous sodium sulfate.

ACKNOWLEDGMENT

It is a pleasure to acknowledge the capable assistance of James Dammicci, Elmer Glaeser and Eileen Weber in the preparation of the dyeings and treatments; to Franklin Dexter for the spectrophotometric studies; to Chester Amick for critically reading the manuscript; to Florence Gustafson for preparing cross sections; to James Leininger for preparing the graphs, and to Bernard Barkowski for making the photographs used as illustrations.

TRADEMARKS

Cyquest is a registered trademark and Calcochrome, Calcofast and Calcomet are trademarks of American Cyanamid Co.

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(1) Eastwood, J A, *Textile Colorist* **63**, 624-625, 646-647 (1941).
(2) Townsend, F, *J Soc Dyers Col* **60**, 144-148 (1944).
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APPENDIX

Standard methods used for this investigation

METHOD NO. 1

Acid Dyeings

2% Calcochrome mordant acid dye
10% Sodium sulfate, anhydrous
8% Ammonium sulfate
Liquor ratio 30:1.
Raise to the boil, boil 30 minutes, add
3% Acetic acid, 28%.
Boil 30 minutes.
Rinse thoroughly.

The average starting pH of 10 dyeings after 10 minutes with wool in bath is 6.45.

The average pH of 10 dyeings at end of dyeing cycle is 4.85.

METHOD NO. 2

Top-Chrome Dyeings

Same as Method No. 1.
Add 1.0% sodium bichromate. Boil 45 minutes. Remove and rinse thoroughly.

The average pH of 10 determinations at end of chroming operation is 5.2.

METHOD NO. 3

Meta Chrome

2.00% Calcochrome mordant acid dye
1.00% Ammonium hydroxide
1.25% Sodium bichromate
5.00% Ammonium sulfate, purified.
Raise to the boil, and boil one hour.
Add 1.5% acetic acid, 28%.
Boil 45 minutes. Remove and rinse thoroughly.
Liquor ratio 40:1.

The average pH of 10 determinations 10 minutes after wool is entered in bath is 7.0.

The average pH of 10 determinations at the end of dyeing cycle is 5.1.

METHOD NO. 4

Bottom Chrome

2.5% Sodium bichromate
2.5% Half-refined tartar (potassium acid tartrate).
Raise slowly to the boil and boil two hours. Rinse thoroughly.
Liquor ratio 40:1.

The pH 10 minutes after wool is entered in bath is 6.1.

The pH at end of bottom-chroming cycle is 6.6.

Fresh Bath

2.0% Calcochrome mordant acid dye
4.0% Ammonium acetate.
Raise to the boil and boil one hour.
Add 1.0-2.0% acetic acid, 28%, to exhaust.
Boil 30 minutes. Rinse thoroughly and dry.
Liquor ratio 40:1.

The pH 10 minutes after wool is entered into bath is 6.65.

The pH at end of dyeing cycle is 5.3.

METHOD NO. 5

Leveling Method for Acid Dyeings

5.0% Acetic acid, 28%
20.0% Anhydrous sodium sulfate.
Liquor ratio 40:1.
Boil one hour. Rinse thoroughly.

The pH 10 minutes after wool is entered into bath is 5.0.

The pH at end of dyeing cycle is 4.8.

METHOD NO. 6

Demetalization and Leveling Bath

8.0% Oxalic acid
20.0% Anhydrous sodium sulfate.
2.0% Cyquest 40 sequestering agent.
Liquor ratio 40:1.
Boil two hours.

The pH 10 minutes after entering wool in bath at room temperature is 2.5.

The pH after boiling two hours is 3.1.

METHOD NO. 7

Rechroming Demetalized and Leveled Dyeings

Fresh bath.
1.0% Sodium bichromate
3.0% Acetic acid, 28%.
Liquor ratio 40:1.
Boil 45 minutes.

The pH 10 minutes after entering wool in bath is 2.6.

The pH after boiling 45 minutes is 3.15.

METHOD NO. 8

Stripping Chromed Dyeings

3% Sodium formaldehyde sulfoxylate (Formapon).
Raise slowly to 150° F. Add
3% acetic acid, 28%.
Raise to the boil and boil 30 minutes.
Fresh bath.
Neutralize with ammonia for 20 minutes at 140° F.
Rinse.

The starting pH (no acid) is 5.7.

The ending pH with acetic acid 28%, 4.55.

1959 AATCC NATIONAL CONVENTION

October 7, 8, 9, 10 • Hotels Sheraton-Park & Shoreham, Washington, DC

ROBERT T GRAHAM
Chairman
Registration Committee
1959 AATCC Convention

ROBERT T GRAHAM, chairman, 1959 National Convention Registration Committee, announces that complete forms for registering for the Convention and hotel accommodations were sent to members of the AATCC before the end of August.

You are encouraged to register as early in advance as possible in order to help the many committees that will make your convention trip more pleasant and profitable and to get the hotel accommodations you would like. YOUR REGISTRATION MUST BE SENT IN BEFORE A HOTEL ROOM AT THE CONVENTION HOTEL WILL BE ASSIGNED TO YOU.

Advance registrations may be cancelled up to and including September 30th by advising the Registration Chairman, Robert T Graham, National Institute of Drycleaning, 909 Burlington Avenue, Silver Spring, Maryland, in writing. Registration fees will be refunded very soon after the Convention is over.

Advance registrants are requested to call at the Convention Registration Desk at the Sheraton-Park Hotel Main Lobby, to obtain their badges, tickets and other convention information, as soon as possible after arriving.

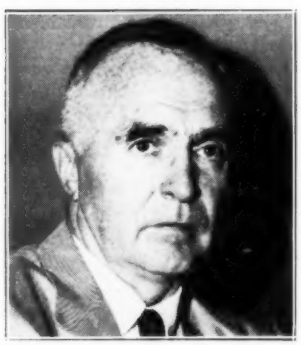
Registration Desk will be open on the following schedule:

Wednesday, October 7—12 noon—9:00 PM
6:00 PM
Friday, October 9—8:00 AM—6:00
Thursday, October 8—8:00 AM—PM

Your convention badge will be required to enter the Exhibition Hall and all meetings of the Association.

The largest registration in the history of the AATCC is expected at the Washington convention. Adequate preparations are being made for registrants from North America and other countries where the textile industry is represented. You are assured a profitable and enjoyable visit in Washington, so prepare now and send your registration and check to cover.

—✦—✦—



JOHN C COOK
Chairman
Printing Committee
1959 AATCC Convention

THE membership at large little realizes the importance of an effective and an efficient Printing Committee chairman. The various forms for registration, programs and countless other matters so essential to a well-run convention, are being capably handled by John C Cook, *Textile Industries*, Atlanta, Georgia.

Mr Cook advises that registration envelopes and hotel registration forms were mailed to the membership in August.

BE SURE TO GET YOUR REGISTRATION FOR THE CONVENTION AND REQUEST FOR HOTEL ACCOMMODATIONS IN THE MAIL AS SOON AS POSSIBLE.

Make checks for Convention Registration payable to:

"1959 AATCC Convention"

GEORGE H LOURIGAN
Chairman
1959 AATCC Convention
Transportation Committee

CONVENTIONEERS attending the 1959 AATCC National Meeting and Exhibition will have no transportation problems after reaching our Capital City, during the Convention period. George H Lourigan, Transportation Committee chairman, who is with Union Carbide Chemicals Company, has arranged for all group transportation required during the Convention. Special plans have been made for transporting the ladies on their visit to the White House. Group transportation will also be provided for the members playing in the Golf Tournament or attending the Maryland-Wake Forest Football Game on Saturday, October 10th. Shuttle bus service will be provided between the two convention hotels (The Shoreham and The Sheraton-Park) on Thursday and Friday mornings as a convenience for the members and guests registered at the Convention.

—✦—✦—

APPROXIMATELY 30 companies have contracted for space in the exhibition hall at the 1959 Washington Convention.

The following firms have taken one or more exhibit spaces:

AATCC
Atlas Electric Devices Co, Inc
Becco Chemical Div
Ciba Co, Inc
Custom Scientific Instruments, Inc
Daily News Record
Diamond Crystal Salt Co
Gaston County Dyeing Machine Co
B F Goodrich Chemical Co
Goodyear Tire & Rubber Co
Hilton-Davis Chemical Co
Hunter Associates Laboratory, Inc
Industrial Rayon Corp
Instrument Development Laboratories, Inc

International Salt Co, Inc
Lestoil, Inc
Mettler Instrument Corp
Morton Salt Co
Nopco Chemical Co
Onyx Oil & Chemical Co
Rohm & Haas Co
Sandoz, Inc
Tanatex Chemical Corp
Testfabrics, Inc
Textile Bulletin
Textile Industries
Textile World
Union Carbide Chemicals Co
Wallerstein Co
Warwick Chemical Div
Jacques Wolf & Co

Other firms wishing to be represented at the 1959 National Convention should contact George P Fulton, Exhibit Chairman, National Institute of Drycleaning, Silver Spring, Maryland, for information on space available. Mr Fulton advises that a few choice exhibit spaces are still available.

The Sheraton-Park Hotel affords the best facilities obtainable to exhibitors and visitors to the Convention. Most of the exhibit spaces are 10 x 10 feet. There are a few larger exhibit spaces still available.

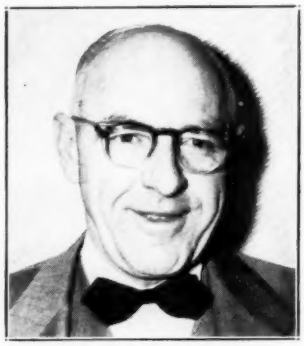
This year's Convention exhibition provides an excellent opportunity for textile firms, and those supplying the industry with raw materials, to display their new products and processes, and for renewing acquaintances with customers and prospects.

RALPH L CARR, chairman of the Reception Committee, 1959 National Convention, announces that the following members will serve on the committee:

Delaware Valley Section	Thomas H Hart Frederick V Traut
Hudson-Mohawk Section	Harvey Gendreau A J Rooney
Northern New England Section	Mrs Geo O Linberg Edward B Bell
Niagara Frontier Section	William H Leyking
Piedmont Section	E P Lavoie Richard E Rettew
Rhode Island Section	Francis H Casey R B Taylorson
Southeastern Section	E R Ravenel
South Central Section	William O Neeb William Quigg
Washington Section	J F Synan (asst chairman)
Western New England Section	A H Rant P W Reeves

The reception committee booth will be near the registration table where a representative will be on hand to give all possible aid to registrants and visitors. It is planned to have a complete list of hotel registrations of all members and guests to assist you in locating your friends that will be attending the largest AATCC Convention and Exhibition in the history of the Association. It is expected that there will be approximately 2200 attending this year's Convention. Adequate hotel and dining services and other facilities to make your Washington visit profitable and enjoyable have been arranged.

RALPH L CARR
Chairman
Reception Committee
1959 AATCC Convention



ARTHUR R THOMPSON
Chairman
Publicity Committee
1959 AATCC Convention

THROUGH the efforts of Arthur R Thompson, Ciba Co, Inc, chairman of the Publicity Committee, the 1959 Convention in Washington has been widely publicized both in this country and abroad. The response to this publicity as well as the general attractiveness of the program are expected to result in the largest registration ever for an AATCC Convention.

All publicity which has appeared in the *Proceedings* to date has been the result of Mr Thompson's efforts.

GOLFERS **FOOTBALL FANS**

You can make reservations for the University of Maryland and Wake Forest Football Game to be played on the Maryland Campus at 1:30 PM Saturday, October 10th, by filling out the form below and returning to John W Howard, chairman, Entertainment Committee, National Cotton Council of America, Ring Building, Room 502, 1200 18th Street NW, Washington 6, DC.

This should be returned before September 24th.

If you plan to play golf Saturday morning, October 10th, for prizes to be awarded under the handicap system, please fill in the following form so that the Committee can make preparations in advance. Tournament play will be on the University of Maryland's new 18-hole "show-piece" golf course. Special transportation will be available for those playing golf and those wanting to go to the football game.

NAME .....

ORGANIZATION .....

ADDRESS .....

I will participate in or attend the following activities:

I. GOLF TOURNAMENT, Saturday, October 10 ☐
Please furnish: Caddy ☐
Caddy Cart ☐
Golf Clubs ☐

II. FOOTBALL, Univ of Maryland vs Wake Forest College ☐
Saturday, October 10 (Seats for this game will be approximately between the 20 and 40 yard lines)
Tickets must be reserved and paid for before September 24.
Please reserve ..... tickets at $4.00 each.

MAKE CHECKS PAYABLE TO
"1959" NATIONAL CONVENTION, AATCC

Activities of the Local Sections

Piedmont

PIEDMONT SECTION will hold its Fall Meeting Sept 25-26 at the Hotel Barringer, Charlotte, NC, at which time J H Hennessey Jr, Polymer Industries, Inc will discuss "The Flash Ager in Action", and Dorel Marian, Francolor, Inc, will speak on "New Developments in the Dyeing of Polyester Fibers and Other Synthetic Fibers in Europe". Both papers will be presented during the Saturday afternoon technical session, beginning at 2 pm.

On Friday afternoon, an organizational meeting of all interested parties in a proposed new Section encompassing northern North Carolina and southern Virginia will be held at 3:30 with E A Murray, secretary pro tem, presiding. This meeting will be held in accordance with Section 2, Article X of the Bylaws.

Two subcommittees of the Section's Research Committee will meet at 9 am Saturday: Subcommittee on Mechanism of Carrier Action (V S Salvin, chairman) and Subcommittee on Mechanism of Chlorine Retention (R L Wayland Jr, chairman). The General Research Committee will meet at 10 am. Basil D Browder, executive vice president, Dan River Mills, Inc, will serve as guest speaker.

Following the Saturday afternoon technical session, the traditional social hour will be held.

Malcolm E Campbell, dean, North Carolina State College School of Textiles, will be the banquet speaker.

Northern New England

NORTHERN NEW ENGLAND Section will hold its 34th Annual Outing on September 25 at the Wachusett Country Club, West Boylston, Mass. This is a change from the previously announced date of September 18.

Cochairmen for the outing, James O Aaronson and H X deRedon, have scheduled a number of events with the cooperation of the following committee members: Basil Nicholson, golf; Hans Walz, horseshoes; Bill Siebert, putting; and John Murphy, baseball.

Tickets for the outing will cost $6.00 per person. This includes dinner, tax, and gratuities but excludes luncheon and golf. Luncheon may be obtained from the club menu.

Southeastern

NONWOVEN FABRICS will be the subject of a symposium to be held September 12 by the Southeastern Section at Ida Callaway Gardens, Pine Mountain, Ga. Master of ceremonies for the event will be Frederick F Hand, sales engineer, Callon Plant, Callaway Mills Co, LaGrange, Ga.

Four papers will be presented.

In the first paper, "Styling and Decorating Acetate Nonwovens" by H W Coates, C E Layman and E L Clements of Celanese Corp of America, an effort will be made to restrict the subject matter to processes peculiar to acetate. The five topics to be treated will be: a) solvent dyeing of acetate nonwovens, b) acetate silk screening and machine printing processes, c) technique for securing three-dimensional effects, d) plasticizer-dye-acetate fiber combinations, and e) acetate fiber molded forms. The paper will be presented by Mr Clements.

D V Probosco, sales mgr, nonwoven fabrics, Wellington Sears Co, will present "A Case Study of Engineering a Nonwoven Fabric". Mr Probosco will select a product in which nonwoven could be used. The steps to be taken to develop a nonwoven fabric which will do the job as good or better than the material being used will be discussed.

D C Nicely, group leader, Nonwoven Fabrics Group, Chemstrand Corp, will discuss "Properties of Acrilan Nonwoven abrics as a Function of Fiber and Binder Content". The physical properties of nonwoven fabrics made from Acrilan fiber bonded with various latices will be described. Different types of binders were used in this study and evaluated as commercially supplied and as compounded with thermoset resin. Data on washability, drycleaning, discoloration, hand, etc will be included. Properties of nonwoven fabrics as a function of fiber length, denier and binder content will be shown.

Paul Homier, supt, Callon Plant, Callaway Mills, in his paper "Areas for Nonwoven Fabrics Advancement", will discuss the needs for improvements in fibers and binders to make nonwovens realize greater gains in volume. He will also discuss the need of help from the conventional textile finishing industry.

South Central

REGISTRATION for the annual summer outing of the South Central Section, held June 26-27 at the Riverside Hotel, Gatlinburg, Tenn, was 164.

In the annual golf tournament, held at Gatlinburg Country Club, James M Stephenson, E T Barwick Mills, Lafayette, Ga, took low gross honors for millmen with an 87, and Thomas J Ratajczak, Berkshire Color & Chemical Co, was the winner among suppliers.

Phillip B Stephenson, Belcraft Laundry, Inc, was the winner in the horseshoe pitching (singles) competition, and W B Mache, Farm & Industrial Chemical Co, won the casting contest.

L to r: Lebron L MaDaris, Crystal Springs Bleachery; Mrs E A Brophy; E V Helms, Geigy Dyestuffs, chairman, South Central Section; E A Brophy, Mohasco Industries, Inc.

The Distaff Side Tries Barber Shop Quartette Singing—Identified are Mildred Haston (left) and Francis Quigg, chairman, ladies activities, third from left.

Friday highlights included a cocktail hour at 6:30 pm, followed by a banquet at 7:30 pm and entertainment with a barber shop quartet contest at 9. Saturday's activities included the golf tournament at 7:30 am, ladies luncheon followed by bridge and canasta at 12:15 pm, horseshoe pitching and bait casting at 2 pm, cocktail hour at 6:30, banquet at 7:30 and dance at 9:30 at the hotel.

In charge of the outing were

HEAD TABLE—SOUTH CENTRAL SECTION OUTING (l to r): Mr & Mrs R J Tyrrell, Peerless Woolen Mills, treasurer; Mr & Mrs W G Quigg, Quigg Chemical Co, outing chairman; E V Helms, Geigy Dyestuffs, chairman, South Central Section; Mr & Mrs Edwin F Jurczak, Burkart-Schier Chemical Co, vice chairman, South Central Section; Mr and Mrs Robert N Ingram; Lebanon Woolen Mills, secretary, South Central Section.

William S Quigg, R J Quigg Chemical Co, general chairman, and Edward G Robinson, Lebanon Woolen Mills, assistant general chairman. Others on the Outing Committee were W G Agnew, Dixie Mercerizing Co, chairman of finance; Nester Groteleuschen, Magnet Mills, Inc, chairman, men's sports; J O Wauford, Lebanon Woolen Mills, chairman, horseshoe pitching; Bill Emerson, Union Carbide Chemicals Co, chairman, fly casting; J T Manning, Standard Knitting Mills, chairman, entertainment; Jack Martin, Geigy Dyestuffs, chairman, golf; Charles Braswell, Standard Knitting Mills, chairman, registration; Mrs William S Quigg and Mrs E V Helms, ladies entertainment; and Betty Ellis, *Daily News Record*, printing and publicity.

Hudson-Mohawk

ONE HUNDRED FOURTEEN members and guests attended the Hudson-Mohawk Section's 11th annual outing and golf tournament held at the Antler's Country Club, Amsterdam, NY, June 26.

Forty-nine members and guests participated in the golf tournament, which saw Carson Thompson of Mohasco Industries, Inc win the Ciba Trophy with a low gross of 69. Among members, Robert Quiri, Ritter Chemical Co, was second, and Arthur W Thomas, A D Julliard Co, third. Among guests, Robert Cox, Solvay Process Div, took low gross honors; Jack Morgan, General Aniline Works, was second; and Richard Ritter, Ritter Chemical Co, third.

Winners of other golfing categories were as follows: Harvey G Gendrau, Sandoz, Inc, longest drive; Jeff Roberts, closest to pin 6; Donald W Reisigle, Mohasco Industries, Inc, closest to pin 16; Mase White, Ritter Chemical Co, most 4s; William Ellsworth, Monsanto Chemical Co, most 5s.

Cochairmen of the outing were William A Nelson and Achilles Mafilios, Ritter Chemical Co. Others on the Outing Committee were Edward A Chevrette, Apex Chemical Co; John J Hanlon and Floyd J Szurek, Mohasco Industries, Inc; and Robert Quiri, Ritter Chemical Co.

HUDSON-MOHAWK SECTION OUTING COMMITTEE (l to r): Edward A Chevrette, John J Hanlon, Achilles Mafilios (cochairman), William A Nelson (cochairman), Robert Quiri, and Floyd J Szurek.

Robert Quiri (left), chairman of the Hudson-Mohawk Section's golf tournament, presents Ciba Trophy to Carson Thompson, Mohasco Industries, Inc, who shot a 69 at the Antler's Country Club course.

HORSESHOES AND DARTS TOURNAMENT WINNERS (l to r): Edward A Chevrette, Apex Chemical Co; Arthur J Bessette, Robert Reis Mfg Co; David O Hamer Jr, Arkansas Co, Inc; Frank Rogan, Cluett, Peabody & Co, Inc; Clayton A Shuttleworth Jr, Mohasco Industries, Inc; Achilles Mafilios, Ritter Chemical Co.

L to r: Dominic J Lambert, Van Raalte Co; Allen Rooney, Geigy Chemical Corp; Michael F Costello, Jacques Wolf & Co; Harry C Spatz, Geigy Chemical Corp; Arthur J Bessette, Robert Reis Mfg Co.

Metropolitan

METROPOLITAN Section will hold its first meeting of the new season on Friday, September 18, 1959 at Geigy Dyestuffs, Division of Geigy Chemical Corporation, Ardsley, NY.

A regular business meeting will begin at 8:00 pm, following which John A Leddy, supervisor—synthetic fibers, coloristic research, Geigy Dyestuffs will present a paper entitled "The I-T Procedure for Applying Cationic Dyestuffs to Acrylic Fibers."

This meeting will be restricted to the first 200 members who pre-register for tickets and will include a dinner and guided tours through Geigy's new installation at Ardsley. These premeeting activities will begin at 6 pm.

Northern New England

THE first technical meeting for the 1959-60 season of the Northern New England Section will be held October 16 at the Lexington Inn, Lexington, Mass. The meeting will concern itself with the permanent setting of wool and the three speakers scheduled for the evening are: Arthur J Farnworth, principal research officer, Commonwealth Scientific and Industrial Research Organization, Belmont, Australia; Gerald Laxer, director of science and technology, The Wool Bureau, Inc, New York, NY; and Harold P Lundgren, chief, Wool and Mohair Laboratories, US Department of Agriculture, Western Regional Research Laboratory, Albany, Calif.

Dr Farnworth will discuss "The Permanent Setting of Wool", which will deal with the theories for the chemical mechanism of permanent set and present the evidence for believing this to involve the rearrangement of hydrogen bonds following the breakdown of some of the disulfide bonds in wool. The speaker will relate this work to its commercial application in the creasing, pleating and flat setting of wool fabrics.

"The Application of Wool Setting Techniques in the Garment Industry" will be the subject of Dr Laxer's paper. Dr Laxer will present a film showing the application of setting solutions and subsequent setting techniques for wool garments and fabrics by the clothing manufacturing industry in the United States. These methods are a direct extension of the durable creasing and pleating process developed by the Commonwealth Scientific and Industrial Research Organization of Australia.

Dr Lundgren will present a paper on the permanent setting of wool.

AATCC Calendar

COUNCIL, COUNCIL COMMITTEES, RESEARCH COMMITTEES
Sept 10-11 (Hotel McAlpin, New York, NY); Sept 10—Council Committees and Research Committees; Sept 11 (AM)—ECR, TCR; Sept 11 (PM)—Council
Oct 7 (2 PM)—Council meeting at Sheraton Park Hotel, Washington, DC

NATIONAL CONVENTIONS
Oct 7-10, 1959 (Sheraton Park and Shoreham Hotels, Washington, DC); Oct 6-8, 1960 (Sheraton Hotel, Philadelphia, Pa); September 28-30, 1961 (Buffalo, NY); 1962 (Southern Region)

DELAWARE VALLEY SECTION
Sept 25 (Hotel Abraham Lincoln, Reading, Pa); Oct 23 (Kugler's Restaurant, Philadelphia, Pa); Dec 4 (Philadelphia, Pa)

HUDSON-MOHAWK SECTION
Oct 2, Nov 20 (Jack's Restaurant, Albany, NY); Jan 15, Mar 27 (Chelsea House, Tribes Hill, NY); May 20 (Ladies Night); June 24 (Annual Outing)

METROPOLITAN SECTION
Sept 18 (Meeting & field trip, Geigy Chemical Corp, Arsley, NY); Oct 16, Nov 20 (Kohler's Swiss Chalet, Rochelle Park, NJ); Jan 15, Feb 19 (Hotel New Yorker, New York, NY); Mar 18, Apr 22 (Swiss Chalet); May 20 (Ladies Night—Swiss Chalet); June 1960 (Outing)

MID-WEST SECTION
Nov 7 (Bismarck Hotel, Chicago, Ill)

NIAGARA FRONTIER SECTION
Sept 25 (Niagara Falls, Ont, Canada); Dec 11 (Annual business meeting, Buffalo, NY)

NORTHERN NEW ENGLAND SECTION
Sept 25 (Outing—Wachusett Country Club, West Boylston, Mass); Oct 16 (Lexington Inn, Lexington, Mass); Dec 4 (Joint symposium with ASME Textile Engineering Div, Smith House, Cambridge, Mass); Jan 15 (Woodlawn Golf Club, Newton, Mass)

PIEDMONT SECTION
Sept 25-26 (Barringer Hotel, Charlotte, NC); Jan 22-23 (Poinsett Hotel, Greenville, SC); April 2-3 (Hotel Robert E Lee, Winston-Salem, NC); June 3-4 (Outing—Ocean Forest Hotel, Myrtle Beach, SC); Sept 23-24, 1960 (Hotel Charlotte, Charlotte, NC)

RHODE ISLAND SECTION
Sept 18 (Management Night); Oct 23 (Prov Eng Soc, Providence, RI); Dec 4 (Annual Meeting—Johnson's Hummocks Grille, Providence, RI)

SOUTH CENTRAL SECTION
Sept 19 (Hotel Patten, Chattanooga, Tenn)

SOUTHEASTERN SECTION
Sept 12 (Ida Cason Callaway Gardens, Pine Mtn, Ga); Dec 5 (Atlanta Athletic Club, Atlanta, Ga)

WESTERN NEW ENGLAND SECTION
Sept 25 (Blake's Restaurant, Springfield, Mass); Oct 23 (Annual Meeting—Rapp's Restaurant, Shelton, Conn); Dec 4 (Hartford, Conn)

Deceased

George A Haigh
Chemist
L B Holliday & Co, Ltd
Deighton, England

Employment Register

This column is open for two insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the secretary of the Association, Post Office Box 28, Lowell, Mass. It is understood that these will be open to inspection by prospective employers who can obtain further information from the secretary.

59-9

Education: BS, textile engineering (US); extra college work in India.
Position desired: Textile engineer.
Location: New York, NY.
Age: 22; single; references.

9-7, 9-21

Future Papers

THE following list is comprised of papers submited to date for publication in future issues of the *Proceedings*. Authors and Technical Program Chairmen are requested to examine this list and call our attention to any omissions.

"Abrasion Resistance of Fabrics: Evaluation of Wear by Means of the Accelerotor"—AATCC Committee on Resistance to Abrasion.

"Analysis of Finish in Textiles"—AATCC Committee on Identification of Finishes on Textiles

"Dimensional Restorability of Woven Textiles after Laundering"—AATCC Committee on Dimensional Changes in Textile Fabrics"

"Dyeing with Direct Dyes—The Effect of Various Salts"—Southeastern Section

"A Study of the Removal of Metal Ions from Cotton Fabric"—Rhode Island Section

"Accelerant Dyeing of Hydrophobic Fibers—Principles and Mechanisms"—Piedmont Section

"The Wet Processing of Cotton/Acrilan Work-Clothing Fabrics"—South Central Section

"THE APPLICATION OF VAT DYES"

Monograph No. 2

American Association of Textile Chemists and Colorists

MONOGRAPH NO. 2, which deals with "The Application of Vat Dyes" was prepared by a board of editors and collaborators in the American Association of Textile Chemists and Colorists. This group represents a complete cross-section of the entire industry so that the text of the Monograph represents information both from laboratory and practical plant standpoints.

The Monograph contains approximately 385 pages with contents of various chapters as follows:

I HISTORY — Natural Indigo and Tyrian Purple; Development of Synthetic Vat Dyestuffs; History of Application of Vat Dyes; The Application of Vat Dyes to Fibers Other Than Cotton.

II PRINCIPLES OF VAT DYE APPLICATION — Types and Forms of Vat Dyes in Commerce; Chemistry of Application; Stripping and Redyeing Faulty Vat Dyeings; Fastness; Print Discharges; Change of Shade in Artificial Light; Dichroic Dyes.

III APPLICATION TO COTTON RAW STOCK — Equipment Used; Methods of Color Application; Typical Examples.

IV APPLICATION TO COTTON SKEINS—Equipment and Methods.

V APPLICATION TO COTTON WARPS—Short Chain Method; Long Chain Warp Dyeing; Continuous Pigment Padding Method.

VI APPLICATION TO COTTON PACKAGES—The Package; Package Dyeing Machines; Chief Factors in Dyeing Packages with Vat Dyestuffs; Dyeing Speed and Levelling Power of Vat Dyes; Dyeing Methods; Oxidation and Finishing; Vat Dyeing on Beams; Illustrative Dyehouse Procedures.

VII APPLICATION TO COTTON PIECE GOODS—History; Jig Methods; Padding Methods; Continuous Methods; Overhead Reel Method; Vat Dyeing of Cotton Narrow Fabrics.

VIII HOSIERY — Application of Colors to Cotton Hosiery; Application of Colors to Mixed-Fiber Hosiery.

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SUBJECT INDEX

From the foregoing, it is seen that this Monograph gives a very comprehensive description of all of the factors involved in the application of vat dyes. In all discussions of application procedure, practical working formulas are given. This publication is of interest to all persons who are either producers or consumers of vat dyes.

The price of "The Application of Vat Dyes" is $5 per copy, postpaid, to members; to nonmembers it is $6 per copy. Quantity discount prices are as follows:

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News of the Trade

8th Chemical Finishing Conference

Attendance at the eighth annual Chemical Finishing Conference to be held in Washington is expected to exceed by far the record attendance of 350 at last year's meeting, according to the National Cotton Council, sponsor of the event.

Nelson F Getchell, technical manager of the Council's utilization research division, says that, because the annual convention of the American Association of Textile Chemists and Colorists immediately follows the Chemical Finishing Conference in Washington, many will take the opportunity to attend both meetings.

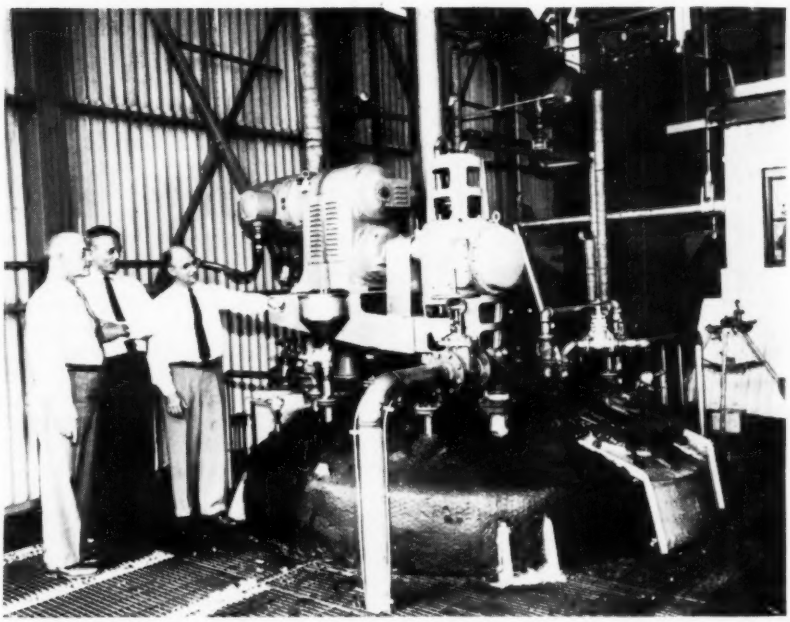
Because of the proximity of the two meetings, this year's conference will be held on one day, October 7, with registration starting at 8:30 am. The AATCC convention will be held Oct 8-10.

Conference general chairman will be Lawrence Marx Jr, president, National Association of Finishers of Textile Fabrics and vice president, United Merchants and Manufacturers, Inc, New York.

Session chairmen are Richard O Steele, head, textile research laboratories, Rohm & Haas Co, Philadelphia, and Lawrence L Heffner, cotton utilization specialist, U S Dept of Agriculture, Clemson, S C.

During the first session the following subjects will be discussed: "Cross-Linking of Cotton at Various Degrees of Fiber Swelling," Wilson A Reeves, Southern Regional Research Laboratory, USDA, New Orleans; "Some Progress in the Cross-Linking of Cotton Cellulose," Giuliana C Tesoro, head, organic research, J P Stevens & Co, Inc, Garfield, N J; and "The Mechanism of Resin Deposition in Cotton," Hermann Rath, German Textile Research Institute, Reutlingen-Stuttgart, Germany.

Following a buffet lunch, the second session will include: "New Developments in Epoxide Wash-Wear Finishes for Cotton," Arnold M Sookne, associate director, Harris Research Laboratories, Inc, Washington, D C; "Performance Characteristics of Chemically Stabilized Knit Cottons," James B Irvine, director, textile process development, Quaker Chemical Products Corp, Conshohocken, Pa; and "Durable Silicone Alloy Water Repellent for Cotton," Charles J Conner, research chemist, SRRL, New Orleans.



Inspecting new $265,000 emulsion kettle at Elizabeth, NJ, plant of Reichhold Chemicals, Inc, are (from left) Stefan H Baum, RCI executive vice president; John G Penniman, manager of the Emulsion Division; and F Albert Smiles, vice president—Elizabeth Plant. Depending on product mix, the new unit reportedly will be able to produce up to 30,000,000 pounds per year of vinyl acetate homopolymer and copolymer emulsions and a full line of acrylic emulsions. One of six RCI emulsion producing facilities in the U S, the new kettle incorporates several unique design features reportedly permitting an optimum balance of properties for the different types of emulsion users.

Fall Meeting, Fiber Society

A two-day program featuring a special report on the textile industry in India, delivery of seven technical papers and election of new officers is scheduled for the Fiber Society's fall meeting at Princeton, N J, Sept 10-11. Sessions will be held at the Textile Research Institute.

The report on Indian textiles will be presented in a banquet address Sept 10 by Helmut Wakeham, staff assistant for research to the chief of operations for Philip Morris Inc, New York. Dr Wakeham sérved two years as director of the Ahmedabad Textile Industry Research Association in India.

The Fiber Society, which meets twice annually, is an organization composed of men and women engaged in research work in fibers, fiber products and fibrous materials, and who have as their objective the advancing of scientific knowledge in those fields. Current officers include S Jack Davis, The Chemstrand Corporation, Decatur, Ala, president; Ernest R Kaswell, Fabric Research Laboratories, Inc, Dedham, Mass, vice president; Julian S Jacobs, Textile Research Institute, secretary; and Hugh Brown, Clemson, S C, treasurer.

Technical papers scheduled for delivery Sept 10 include "Dynamics of Textile Materials," by D R Petterson, G M Stewart, and F A O'Dell, of Army Chemical Center, Md; "The Failure of Strong Fibers Under Repeated Loading," by J L Riley and W George, Celanese Corp of America, Summit, N J; and "Effects of Drawing and Skin: Core Ratio on Properties of Viscose Rayon Filaments," by J H Dusenbury, C Danziger and E Dey, of TRI.

Continuing papers on September 11 will be "The Physical State of Direct Dyes in Viscose and Its Influence on Lightfastness" by L Weissbein and G E Coven, American Cyanamid Co, Bound Brook, N J; "Modification of Fibers by Grafting" by E H Immergut, Dunlop Research Center, Toronto, Canada; "Lubrication of Fibers and Drafting Properties of Roving," by G H Lourigan and D M Cates, North Carolina State College, Raleigh; and "Heat Transmission Through Fibrous Cotton Slabs" by H J Janssen and R V Bailey, Southern Regional Research Laboratories, USDA, New Orleans, La.

ROSCC Names Canadian Sales Organization

Richmond Oil, Soap & Chemical Co, 1041 Frankford Ave, Philadelphia 25, Pa, has announced the appointment of Fawcett & Co, Ltd, 34 High Park Blvd, Toronto 3, Ontario, as its sales and service organization in Canada. Fawcett & Co, Ltd, headed by Noel Fawcett, will sell and service the various textile chemical specialties manufactured by the Richmond Oil, Soap & Chemical Co.

Construction Begun on Du Pont's Caprolactam Plant in Texas

Construction has started on the Du Pont Company's new, 50,000,000-pound-a-year plant to manufacture caprolactam, an intermediate chemical for nylon 6 fiber and plastics.

The multimillion dollar plant is expected to be completed and ready to start operations by the end of 1960. It is being built on the company's present plant site in Beaumont, Texas, and use of this site with existing utilities and other facilities will expedite completion of the caprolactam unit, it is stated.

Provision for a substantial increase in capacity above 50,000,000 pounds has been incorporated in the plant design. The caprolactam and a co-product, cyclohexylamine, will be produced primarily for sale outside the company.

The Beaumont plant reportedly will produce caprolactam by a new process made possible by technological improvements resulting from a lengthy research effort and the use of low-cost petroleum derivatives.

Dow-Corning Office Move

The new location for two sections of Dow Corning Corp's New York sales office was announced last month. Fiber Finishes Sales and Service and the International Department have established their respective offices in the new Corning Building at 717 Fifth Avenue, New York 22, N Y.

The Fiber Finishes Sales and Service group is responsible for the New York Area technical service and promotion of Syl-mer grade silicone textile finishes and Sylflex silicone treatment for leather. The new offices reportedly will have facilities for exhibiting the latest examples of finished fabrics, garments and treated shoes for the convenience of mills, finishers, manufacturers and retailers.

Morningstar-Paisley Acquires New Potato Starch Plant

Acquisition of the capital stock of the San Luis Valley Starch Co and the Monte Vista Sales Co, Monte Vista, Colo, was recently revealed by Morningstar-Paisley, Inc, New York, NY. The acquired firm produces potato starch from the russet type of potato grown in large volume in the San Luis Valley of Colorado.

Physical assets of San Luis Valley Starch Company consist of a factory building equipped with modern processing equipment and warehouse facilities. Capacity of the plant is estimated at ten million pounds per processing season, which extends from September to approximately April or May. The plant was built in 1954 and has been operated by Robert H Detweiler, who will continue as manager of the plant under the new ownership.

Morningstar-Paisley now operates three starch plants in Aroostook County, Maine, two at Houlton, Maine, and one at Mars Hill, Maine. Addition of the San Luis plant reportedly will provide diversification of production, continuity of raw material supply, increased storage facilities, and a more centralized shipping point for better service to Mid-Western, Southern and West Coast markets.

A portion of the output will be processed over hot rolls at the firm's Haberland Division in Clifton, N J, to produce pregelatinized starch.

Sound Film of New Permanent Creasing Process Produced by Wool Bureau

A 16-mm sound film of a permanent creasing process for men's wool trousers has just been completed by the Wool Bureau.

Running approximately 15 minutes, the film will be sent out on loan to trade groups and manufacturers who may use it to study the variety of equipment and techniques developed for the process.

An example of new factory technology perfected for the process is the operation of three separate presses by a single operator. This is shown in two different pressroom layouts with operators using regular presses which have been modified for automatic control, and also newer presses which control automatically the various pressing operations and the timing for each step.

Copies of the film may be obtained by writing the Department of Science and Technology at the Wool Bureau, 360 Lexington Avenue, New York 17, NY.

Production Started at NSCC Vinyl Emulsion Polymerization Plant

National Starch and Chemical Co (Canada) Ltd last month announced the start of production at its newly completed vinyl emulsion polymerization plant at Toronto, Ont.

B V Schaub, president, said that initially the new plant will produce vinyl copolymers and homopolymers in emulsion form, and is built to allow for future increase in capacity.

Production from the new plant will be used both internally for the manufacture of adhesives—a field in which the company is well known—and for sales throughout Canada to manufacturers in the textile and other fields.

National Starch and Chemical Corporation (formerly known as National Starch Products Inc) is the parent company, with operations in England, Mexico, and the USA, as well as Canada.

American Conditioning House Renamed

American Conditioning House, Inc, Boston, Mass, will now be known by the name ACH Fiber Service, Inc. This change is said to be in harmony with the broadened scope of the organization's activities and staff facilities.

American Conditioning House, Inc was organized at the close of World War II to sample and evaluate wool in the commercial markets of the United States. The original name of the corporation was taken from the conditioning houses in Europe, which were identified with reliable wool evaluation by laboratory tests. This association with one fiber type and traditional testing concepts now has become too limited, in view of current expanding activities of American Conditioning House, Inc.

The firm has met interest in evaluating specialty fibers according to standards developed in the trade, economic changes that require more rigid cost and quality control, and demands for fiber services in connection with the new textile fiber products labeling laws by recruiting new staff members, who are authorities in their special fields. In addition, the organization is currently engaged in new processing studies on fibers in general, without limitation to wool.

Recent senior staff appointments include Samuel J Golub as associate laboratory director and Herbert C Haller as assistant to Herbert Wollner, president. In addition, the consulting and advisory services of a number of recognized authorities in supplemental fields have been retained.

Century Chemical Acquires Control of Wilson Organic Chemicals

Century Chemical Corp, 60 E 42nd St, New York 17, N Y, has acquired controlling interest in Wilson Organic Chemicals, Inc, Sayerville, N J. The transaction, completed on July 31st, gives Century over 80% of Wilson's outstanding stock. The company will be operated as the Wilson Organic Chemicals Division of Century Chemical Corporation.

Wilson is reputedly the largest independent U S manufacturer of phthalocyanine dyes and pigments. The company also manufactures aromatic fine chemicals and intermediates for the drug and pharmaceutical industries.

Century Chemical reportedly will initiate, almost immediately, an extensive modernization and expansion program of Wilson's manufacturing facilities. Sales last year were said to be approximately three-quarters of a million dollars and Century hopes to triple sales in 1960.

GAF Appoints New Ga, Fla Distributors

General Aniline & Film Corp has announced the appointment of Atlanta Solvents & Chemical Co, 2323 Lanasol Rd, Doranville, Ga, and Moss Soap Co, 1157 NW 22nd St, Miami, Fla, as distributors for Antara Chemicals, a sales division of GAF.

Hodag Chemical Opens Expanded Facilities

Hodag Chemical Corp, 7247 Central Park, Chicago, Ill, has completed construction of enlarged production, laboratory and office facilities, which reportedly will allow the company to handle the increasing demand for its line of antifoams, esters, emulsifiers, flocculating agents, and other surface-active chemicals.

The new facilities include stainless-steel blending tanks and a sonic homogenizer for the manufacture of silicone antifoams and other silicone products.

The construction has quadrupled Hodag's former plant space. Laboratory space has been tripled and office space doubled. Also, Hodag has added laboratory personnel and equipment as well as increased warehouse space for storage of raw materials and finished products.

KixMiller

Brooks

Celanese Organizes Three New Companies

Celanese Corporation of America has organized three new companies to administer the manufacture and marketing of its fiber, chemical and plastics products.

The companies, Celanese Fibers Co, Celanese Chemical Co and Celanese Plastics Co, will function as operating divisions of the parent firm. They succeed the former Celanese fibers, plastics and chemical divisions.

John W Brooks has been appointed president of Celanese Fibers Co and Richard W KixMiller have been named president of Celanese Chemical Co and also of Celanese Plastics Co. Both Mr Brooks and Mr KixMiller will continue to serve, in addition, as vice presidents of Celanese Corporation of America, of which Mr KixMiller also continues as a director.

It was emphasized that, although both the Celanese chemical and plastics companies are headed by Mr KixMiller, the two organizations will continue to function separately and independently.

$100,000 in Government Contracts for LTIRF

Government contracts amounting to nearly $100,000 were signed recently by the Lowell Technological Institute Research Foundation.

The Air Force Cambridge Research Center has authorized two six-month projects each at a cost of $30,000. Research directed toward the design and development of a mobile optical and infrared calibration laboratory will be supervised by Louis C Block of the LTI faculty, project leader. The second project, with Kenneth F Kinnard as project principal, is for research directed toward the design, development, construction, and operation of a prototype radiometric space-vehicle tracking station.

An eight-month project amounting to $3422, sponsored by the Quartermaster Research and Engineering Command, concerns a study of the various methods of producing mats for the evaluation of thermal protection of natural and synthetic fibers. This will be directed by Norwood H Keeney of LTI's paper engineering department.

As reported previously, the U S Department of Agriculture is sponsoring a three-year project of $35,037 for investigations designed to develop winter-weight cotton fabrics having good warmth properties using the woolen system for producing the yarns. This is being conducted under the direction of Emery I Valko, professor of chemistry at the Institute.

Fiber Industries, Inc Licensed to Produce Teron in US

A license to produce and market a polyester fiber in the United States before expiration of the basic patent in July, 1961, has been granted to Fiber Industries, Inc, by E I duPont de Nemours & Co, Inc.

Announcement of the licensing was made recently by James H Black, president of Fiber Industries, Inc, a company jointly owned by Celanese Corporation of America and Imperial Chemical Industries Limited of Great Britain.

At the same time, Mr Black reported plans by Fiber Industries to market commercial quantities of its new polyester fiber, Teron, by the middle of 1960. The fiber will be produced in a plant now under construction in Shelby, NC, in both staple and filament forms, for primary end uses in apparel and home furnishings. It will be marketed by Celanese.

The polyester fiber, known chemically as polyethylene terephthalate, is claimed to be one of the fastest-growing synthetic textiles. It was developed in 1941 in the laboratories of Calico Printers' Association, Ltd, in Great Britain. Rights to manufacture and market the fiber in the United States were sold by Calico Printers to DuPont.

Fiber Industries' Shelby plant reportedly will have an ultimate capacity of 40 million pounds a year.

THE AATCC CONVENTION NUMBER WILL BE PUBLISHED SEPTEMBER 21

First Fiber-reactive Dyes for Nylon

Arnold, Hoffman & Co, Inc, Providence 3, RI, has released the initial four members of a new range of fiber-reactive disperse dyestuffs for dyeing nylon. Known as the Procinyl Dyestuffs, these patented specialties have been invented and developed by Arnold, Hoffman's parent company, Imperial Chemical Industries Ltd of England, manufacturers of the Procion reactive dyes.

According to Arnold, Hoffman, the new Procinyl dyestuffs, when applied to nylon and other polyamide fibers, display the exceptionally desirable attributes of established disperse dyes. Namely, they are said to exhibit good leveling, good coverage of irregular dyeing yarns, good compatibility in admixture, and excellent penetration coupled with wetfastness properties considerably in excess of those normally associated with the disperse range.

Application conditions are reported to be simple. Dyeing is begun from a weakly acidic dyebath, which is made alkaline towards the completion of the dyeing cycle, thereby making certain groups in the Procinyl dye molecules react with the amine or amide groups of the polyamide fiber, so as to produce dyeings with high wetfastness properties.

It is reported that all forms of nylon and other polyamide fibers of both the staple fiber and continuous filament types—and including woven piece goods, knitted goods, and hosiery—are dyed satisfactorily with Procinyl dyes, which may be applied on the jig, on the dye kettle, and in the paddle machine. Although it is thought that the initial interest will be covered by batchwise piece goods application, the possibility of continuous and semicontinuous piece goods dyeing operations is also a consideration. Stock dyeing and yarn dyeing using circulating machines represent further potential processes for these new dyestuffs.

The initial four Procinyl dyes, a yellow, an orange, a scarlet, and a blue, are said to permit an extensive range of shades to be produced because of wide intercompatibility of the new dyes. With the possible exception of yarns and fabrics for which exceptionally stringent standards of lightfastness have been set, it appears that Procinyl dyes will meet the requirements for a wide variety of hosiery, lingerie and wearing apparel where high wetfastness properties are required. It is also suggested by Arnold, Hoffman that, at the same time, the dyer can be relieved of some of his current problems associated with the dyeing of barré nylon, particularly when bulked yarns are being used.

Being disperse dyes, the Procinyl dyes will color other synthetic fibers such as acetate, triacetate, polyesters, and acrylic fibers; but with the exception of the unmodified acrylics, no reaction with the fiber takes place and accordingly the resultant dyeings have similar fastness properties to the well-established disperse dyes, it is claimed.

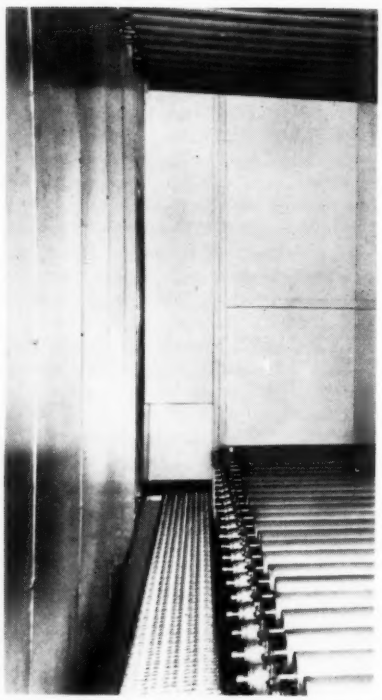
In the case of Procinyl Yellow GS, it is reported, outstandingly high lightfastness and resistance to heat treatments are realized on all polyester fibers.

The announcement of the Procinyl dyes comes during the 20th anniversary year for nylon, at a time when nylon is outstripped only by rayon in total pounds of man-made fibers produced.

New Curing Machine for Resin Finishes

A new roll-type curing machine for resin-type finishes has been developed by Adams-Zeller Corp, Glenside, Pa.

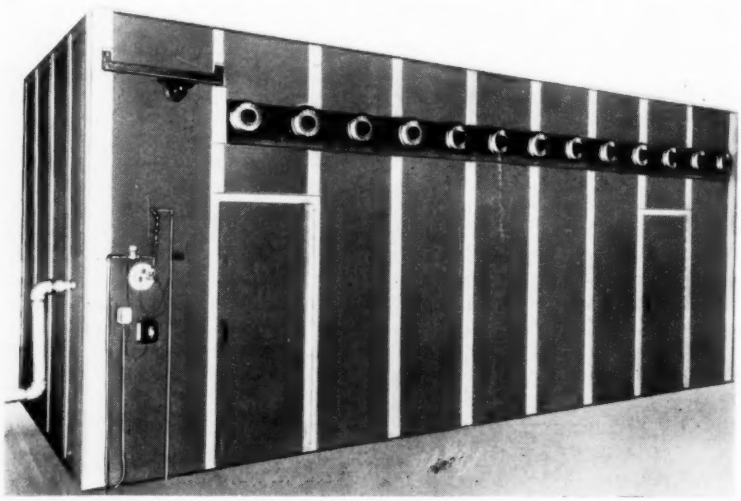
The first installation of the new machine has been made at Allied Textile Printers, Paterson, N J, where it reportedly operates in a finishing range at 60 to 65 yards per minute.



Curing chamber interior showing catwalk at floor level which provides accessibility for threading-up, adjusting, and cleaning.

After drying on a tenter frame, the resin-treated cloth enters the Adams-Zeller machine where it is cured in approximately three minutes. The cured goods are delivered to a rotary cutter-winder for wind-up.

In the Adams-Zeller machine, centrifugal blowers discharge air heated



Adams-Zeller roll type curing machine installed at Allied Textile Printers, Paterson, NJ. Seal-type gasketed hinged doors provide access to curing chamber.

by gas burners into a plenum chamber. The heated air is forced through adjustable air orifices above each roll. It is claimed that, through accurate orificing, even temperatures are maintained at each roll for proper curing. Constant tension is maintained as the cloth moves through the machine.

At Allied Textile Printers, the Adams-Zeller machine was erected in five days. The machine was pre-erected in the Adams-Zeller shops, disassembled, and match-marked.

The number and size of rolls used in the Adams-Zeller machine are determined by the particular application. At Allied Textile Printers, the unit has 79 4-inch stainless-steel rolls with 72-inch face. With this arrangement, a total of 195 yards of cloth is in the machine at one time.

A bulletin on the new roll-type curing machine is available from Adams-Zeller Corp on request.

Conoco DBCL

The introduction of a new petrochemical product, Conoco DBCL, was announced recently by Continental Oil Company.

Conoco DBCL is an intermediate for use in the manufacture of cationic and nonionic detergents. It reportedly has a minimum dodecylbenzyl chloride content of 90 percent and is light straw colored, with a specific gravity of approximately 0.965.

The new petrochemical product is said to be easy to handle and to readily react with tertiary amines to form cationic surface-active agents, ie, quaternary ammonium salts. The quaternary finds applications as the active ingredients in sanitizers and antistatic agents for fibers, paper and plastics. If the Conoco DBCL is reacted with polyglycols, nonionic surface-active materials reportedly are formed.

Rezosol 1460

A new resin binder for sizing Dacron-cotton warps is now available from E F Houghton & Co. Called Rezosol 1460, this new polymer is supplied as a liquid and reportedly can be added without special precautions when the size is prepared.

Among the advantages of Rezosol 1460 reported by the supplier are kettle cost reduction of over $12.00 (per 200-gallon batch of size), reductions in seconds and shedding, improved separation, and up to 100 more yards of warp per beam.

A large southern mill weaving a 65% Dacron-35% cotton blend is claimed to have pushed weaving efficiency from 83% to 94.6% during a field trial of the new binder. During this period seconds dropped from over 7% to under 3%, it is reported.

The supplier has prepared a data sheet which describes Rezosol 1460.

NAMES IN THE NEWS

Van Wirt

Imperial Color Chemical & Paper Corp, Glens Falls, N Y, has announced the election of **Alfred E Van Wirt** as executive vice president. Mr Van Wirt is currently vice president and technical director.

Mr Van Wirt joined Imperial in 1925 as a production staff assistant. In 1935, Mr Van Wirt was named technical director in charge of all research laboratories, as well as engineering and technical operations. He was elected to the board of directors in 1946 and became vice president in 1953.

F Dean Hildebrandt has been elected an executive vice president of McKesson & Robbins, Inc.

Mr Hildebrandt joined the company in 1945 as general manager of McKesson's newly formed chemical distributing organization. In 1955, the company's chemical business was reorganized as an independent department, and Mr Hildebrandt was elected a vice president.

Willingham

W Bernard Willingham, who has piloted a tremendous equipment and sales expansion program at Ohio Falls, Inc, since he joined the Louisville, Ky, firm in 1952, became the company's new president on September 1.

Willingham's purchase of stock owned by former president **William I Wymond** gives him 100% ownership of the dyeing and finishing company, which has been processing and dyeing for canvas goods manufacturers since 1897.

Ohio Falls' new president joined the firm July 1, 1952, as executive vice president. He had been executive vice president and general manager of Ballard & Ballard in Louisville, now a subsidiary of Pillsbury Mills.

Burton Blagman, formerly a developmental chemist for Princeton Knitting Mills, Watertown, Conn, has been named assistant director for chemical development for Malden Mills, Inc, Lawrence, Mass.

Moody

Putnam Chemical Corp, Beacon, N Y, at a recent meeting of the board of directors, elected **Leon E Moody** executive vice president of the Corporation and re-elected **Fritz G Naumann** President. **Richard E Metz** was elected chairman of the board.

Mr Metz is active in banking circles and holds various directorships in the chemical and allied industries.

Mr Moody has been active until recently as plant manager of the Dublin, Va, Dyeing and Finishing Plant of Burlington Industries. Previous to that he was associated in similar capacities with Duplan Corp in Canada, U S Finishing Co in New England, and with American Cyanamid Co as technical manager of its textile resin department.

Charles L Kale, Mt Holly, NC, has been named recipient of the Dickson Scholarship at North Carolina State College. The scholarship is valued at $750 a year, or $3,000 for the entire four years.

Brouillard

Webb

The appointment of **Robert E Brouillard** as sales manager—pigments, and **H Alison Webb** as sales manager—dyestuffs, has been announced by the Dyestuff and Chemical Division of General Aniline & Film Corporation.

Dr Brouillard, after several years with American Cyanamid Co, joined GAF in December 1948 at the Linden plant and moved to New York as assistant manager of the Pigment Sales Department in February 1957. In January 1958 he was named product manager—pigments.

Mr Webb joined the company in 1935. After serving in both technical and sales capacities in the Charlotte office for many years, he was appointed manager of the Chattanooga office when it was opened in January 1954. He came to New York as assistant sales manager, dyestuffs and pigments, in October 1957.

Diamond Alkali Co last month announced the promotion of **R E Shepard** and transfer of **W E Downey** in its Electro Chemicals Division, Technical Service Group.

At his new post, Mr Shepard will assume responsibilities as a field technical service man. Previously, he worked in the Company's plant control laboratories as well as various research laboratories before joining the Technical Service Group in 1958. He has been with Diamond since 1940.

Mr Downey transfers to the vacancy created by Shepard in the Technical Service Group. For the past three years he has been a member of the Analytical Division in the Company's Research Department.

Appointment of **Maurice S Letourneau** as New York District sales manager for the Polyco-Monomer Department of the Borden Chemical Co has been announced.

Mr Letourneau will be responsible for sales of Borden's full line of polymers for the textile, paper, paint, and adhesives industries in the Metropolitan New York area. He previously served the department for almost two years as technical sales representative in an area encompassing the Carolinas and Virginia. Prior to that he covered the New England states.

Donald H Loeber, former executive of Catalytic Construction Co, has joined W F Fancourt Co, Philadelphia, Pa, as general office manager and assistant to the president, **John L Fancourt.**

Before joining the Fancourt company, Mr Loeber was manager of systems and procedure, and assistant manager of general accounting for Catalytic Construction.

Thies

Kelly

Two new sales departments have been established within the Chemical Division of The Goodyear Tire & Rubber Company.

The new departments, Polyester Products and Adhesives, join with the Rubber and Rubber Chemicals, Coatings and Plastics Departments to bring the division's total sales units to five.

Appointed as manager of the Polyester Products Department was **L P Thies,** former head of the division's Detroit office. In his new post, Thies will be responsible for sales and technical sales service of Goodyear's polyester resin, Vitel, to the fiber and textile industries.

W E Kelly, former manager of the division's St Louis office, was named to head up the new Adhesives Department. In this capacity, he will work with **R S Sanders,** manager of Pliobond sales, to coordinate the sales activities of the division's entire line of adhesive materials.

Appointment of **M James Campbell** to position of assistant to the vice president of Allied Chemical's Solvay Process Division, was announced last month.

Succeeding Mr Campbell as manager at Syracuse, Solvay's largest plant, will be **Robert B Lautner,** currently manager of Solvay's Moundsville, W Va operations. And moving to Moundsville as manager will be **Robert E James** who has been manager of Solvay's Baltimore, Md, plant. With the assumption of greater responsibility by Mr James at Solvay's Moundsville plant, the Baltimore plant will be under the direction of **Edward J Walsh,** assistant manager.

Bunting

Welsh

Sandoz, Inc has announced the addition of **Richard S Bunting** to its dyestuff division sales force and the transfer of **Robert Welsh** from a dyestuff sales assignment in the Philadelphia territory to the Pacific Northwest area.

Mr Bunting, formerly sales mgr, Special Products Div, Russell Mfg Co, has been assigned to the Hudson, Mass, office, reporting to Robert Schaefer, New England District sales manager. Mr Bunting's experience in the textile industry also includes employment in technical service and as divisional sales manager with Pacific Mills/Burlington Industries.

The announcement of Mr Welsh's transfer to the Pacific Northwest was made in July, exactly 40 years after Sandoz opened for business in New York with one man and a secretary to cover the entire country. Sandoz now operates laboratories and sales offices in six other textile centers in the United States.

The appointment of two new branch managers, a regional engineer and two new sales engineers has been announced by The Foxboro Company, Foxboro, Mass.

R F Sutton was named branch manager at Philadelphia; **T A Jones,** branch manager of the Atlanta office; **F H Leathers,** regional engineer for Southeastern states; **G W Howlett,** sales engineer at Pensacola, Fla; and **L L Hodges,** sales engineer at Mobile, Ala.

Mr Sutton moves to his Philadelphia assignment from the Pensacola office where he has served as sales engineer since 1956.

Mr Jones has been a sales engineer at the Charlotte, NC, office since 1955.

Mr Leathers, who will assist Foxboro application engineers in planning instrument projects in the Southeast, has been with the Company since 1948.

Transferring from their sales engineering positions in the Atlanta office are Mr Howlett, who will serve customers in the Pensacola area, and Mr Hodges, who will conduct the sales activities of Foxboro's new office in Mobile.

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Herard

Richard A Herard, for nine years with David Gessner Co, manufacturer of cloth finishing machinery, Worcester, Mass, in the departments of engineering, manufacturing, and sales, has been appointed Southern representative of that company. He will be in charge of the Gessner office, 222 Piedmont Building, Greensboro, NC, which was opened in 1954.

Paul F Riedl, Gessner service representative based in Greensboro, will continue in his capacity and with headquarters at the same address.

G Preston Hoff, manager of the Planning Division in the Du Pont Company's Textile Fibers Department, retired at the end of July after 30 years with the company.

Prominently identified with synthetic fiber research and development since 1925, Dr Hoff pioneered the early development work on high tenacity rayon and selected the name Cordura for the yarn. Cordura was the first successful high tenacity rayon yarn for use in automobile tires.

Dr Hoff also was in charge of the research group responsible for carrying the nylon development from the laboratory stage to commercial manufacture. Later he directed research leading to the commercialization of Orlon acrylic fiber.

Wayne A Sisson, American Viscose Corp, has resigned as chairman of Textile Research Institute's General Research Advisory Committee. He is succeeded in the post by **Norman C Armitage,** Deering Milliken Research Corp, formerly vice chairman of the Committee. **Edawrd W Lawrence,** Cranston Print Works Co, was elected to serve a two-year term as vice chairman.

Richard W Pease has been appointed director of engineering and manufacturing by Polyvinyl Chemicals, Inc, Peabody, Mass. His appointment includes responsibility for Polyvinyl's current plant expansion program.

Pease joins Polyvinyl from the Borden Chemical Company's Polymer Division, where he was production manager.

Gaylord

Norman G Gaylord has been elected vice president, research and development, at the Polymer Division of Western Petrochemical Corp. Dr Gaylord was formerly assistant director of the organic chemistry department at Interchemical Corp.

Dr Gaylord is senior lecturer at the Polytechnic Institute of Brooklyn, and has served as adjunct professor of chemistry at Canisius College, Buffalo, NY.

Dr Gaylord has published two books and many articles and papers in the field of high polymer chemistry. His latest book, co-authored with Herman Mark, is titled "Linear and Sterioregular Addition Polymers: Polymerization with Controlled Propagation."

Organizational changes designed for more effective handling of its expanding interests in coatings for the textile industry have been announced by the Dow Chemical Company.

L H Silvernail has been promoted to group manager in the company's Coatings Technical Service, in charge of four industry sections, including the textile section, which is concerned with Dow plastic products going into the textile industry.

In active charge of the textile section is **J P Strasser,** who was advanced from the post of customer service chemist in the textile field.

Ralph Hand, formerly in charge of this section, has taken over the CTS Building Products section.

Silvernail has been head of the paper section since 1952.

Strasser's entire service with Dow has been devoted to textile activities.

George W Pierce, formerly executive director and general manager of American Efird Mills, Finishing Division, recently joined Riggs & Lombard and its affiliated companies, Cook Machine Co and Parks & Woolson Machine Co, as Southern sales engineer.

In his new capacity, Mr Pierce will serve the cotton and synthetic dyeing and finishing field in the South, and make his headquarters at the Riggs & Lombard Charlotte, NC, office.

Simonian

John V Simonian has been appointed to the research and development staff of Crown Chemical Corp, Providence, RI, as director of the extended resin research program.

Mr Simionian has served as a research chemist with the Dow Chemical Company, the Geigy Chemical Corporation, and the Warwick Chemical Company, a Division of Sun Chemical.

While with Warwick Chemical, Mr Simonian was a group leader in textile research.

Paul A Dunkel & Co, Inc, Jersey City, NJ, importers and processors of vegetable gums, has named **Charles M Ferri** sales and technical director. Mr Ferri will be in charge of quality control, new product development, and technical service.

Prior to Joining Paul A Dunkel & Co, Mr Ferri was affiliated with Thurston and Braidich for 15 years as technical director.

Six employees of the Minnesota Mining and Manufacturing Co, St Paul, Minn, recently attended a company-sponsored textile workshop at Lowell Technological Institute, conducted by **John J McDonald,** head of the Institute's finishing department. They were **Bart Corser, Sidney M Leahy, John L Rendall, Allan E Reynolds, Richard G Riedesel,** and **Charles Tanguary.**

The professional development seminar was concerned with all phases of textiles, and the instructional staff assisting Prof McDonald included **Jacob K Frederick Jr, Edward L Golec,** and **Fritz F Kobayashi.**

James M Willhoite has joined the new product applications department of the research laboratories of Armour Industrial Chemical Company.

Mr Willhoite was formerly associated with Mallinckrodt Chemical Works as an analytical chemist.

In his new position, Mr Willhoite will be responsible for obtaining the physical and chemical properties of new products prepared by the organic research section.

Joseph Bancroft & Sons Co, Wilmington, Del, has announced five staff changes in the Engineering Section, Chemical Research and Products Division, which handles the Ban-Lon program.

J D Bradley has been named an assistant group leader in the Textile Group, in charge of quality control evaluation. **A R Benedek** has been transferred from the Mechanical to the Textile Group as testing supervisor, reporting to Mr Bradley.

F H Hartgrove, assistant group leader, has been placed in charge of activities involving trademark usage, labelling specifications, etc.

G N Tillottson and **Alice Goold** have been placed in charge of all new test methods and specifications.

Several appointments in technical service were recently announced by Union Carbide Chemicals Co, division of Union Carbide Corp. **John R Anderson, Carl K Fink, Samuel M Livengood, William H Millett,** and **Edward R Weidlein Jr** have been appointed assistant directors of the Technical Service Laboratory; **E Lacy Gibson** has been named administrative assistant to the director. The five assistant directors will be responsible for experimental programs and business functions of the laboratory now being built near Tarrytown, NY, and due for completion in late 1959 or early 1960. Mr Gibson will co-ordinate and administer certain of the use research, customer service, and training functions.

Drs Anderson, Fink, and Livengood and Mr Weidlein are senior fellows at Mellon Institute, Pittsburgh, Pa.

Dr Millet, since 1958, has been serving as technical consultant to the New Chemicals Dept.

Mr Gibson has been serving as Indiana area coordinator for college recruiting since 1958.

Creation of a new Advertising and Publicity Department for Food Machinery and Chemical Corporation's Chemical Divisions has been announced. The Chemical Divisions of FMC are Westvaco Mineral Products, Westvaco Chlor-Alkali, Becco Chemical, Chemicals & Plastics and Niagara Chemical. The new Department will provide services to each of these Divisions (and to appropriate staff departments) in the areas of advertising, technical and sales literature and product and personnel publicity.

Named as manager of the Department is **Desmond M C Reilly,** manager of advertising and technical information of FMC's Chemicals & Plastics Division since 1957.

Named as assistant manager (publicity) in the new Department is **Robert B Rumely,** formerly FMC Chemical Divisions' public relations representative.

Don Hanson, named as assistant manager (advertising) has been assistant advertising manager with FMC's Niagara Chemical Division since July 1958.

The appointment of two new members to the sales staff of Bradford Dyeing Association, New York, N Y, has been announced. **Joseph Brown,** former service manager of Bradford's New York office, joined the sales staff on July 1.

Allan Ralston, a former Bradford employee, returned to the company on August 1 as a member of the sales staff. Ralston was associated with Bradford Dyeing Association from 1947 to 1954, both in the laboratory and the New York office, and left the organization to go into business for himself.

Raymond Corder has been appointed manager of Creative PR, Inc, public relations subsidiary of Anderson & Cairns, Inc, advertising agency. He succeeds **Ashley W Burner,** who has resigned.

Mr Corder, formerly assistant director, will be responsible for coordinating the public relations and publicity activities for both Anderson & Cairns clients and nonclients.

A three-member executive committee of Century Chemical Corp has been elected by the Company's board of directors. The committee members are **Theodore S Hodgins,** president of Century; **George T Bayley,** chairman of the board of Calkin & Bayley, Inc, industrial consultants; and **Charles W B Wardell Jr,** vice chairman of Deltec Corp, international financial consultants.

OBITUARY

W C DURFEE

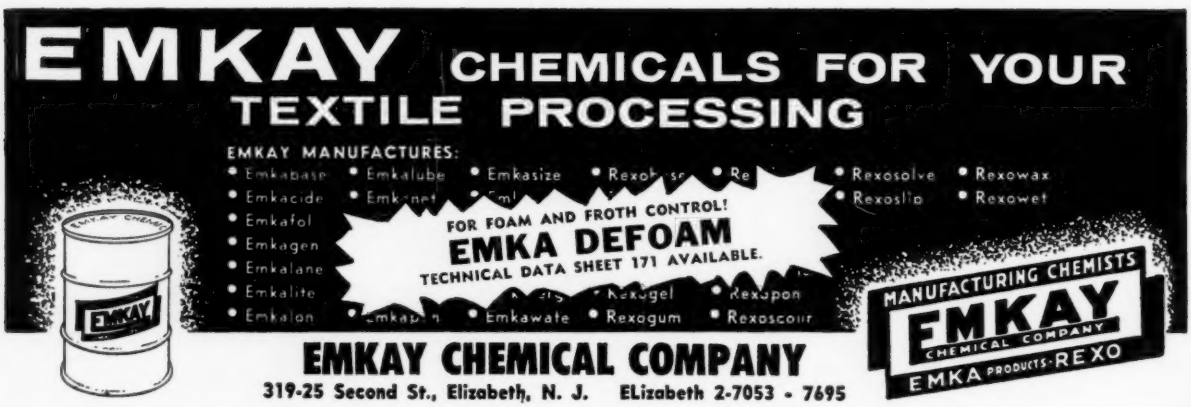
WALTER C DURFEE died in Jamaica Plain, Mass on August 14. Until his retirement, he was president of the W C Durfee Co, founded by his father, Winthrop C Durfee.

Mr Durfee was a charter member of AATCC. For many years he was a recognized authority on colorfastness to crocking, having developed the Crockmeter and assigned his patent to AATCC. This has been largely accepted as part of AATCC's Standard Test Method abroad as well as in this country, and has been sold at profit of several thousand dollars in royalties to the Research Fund of the Association.

He was a graduate of the Roxbury Latin School, and of Harvard College, Class of 1904. He is survived by his wife, Elizabeth Davidson Durfee, three daughters, seven grandchildren, and his brother and sister.

C L COBLE

CHARLES L COBLE, production manager of Apex Chemical Co, Inc, Elizabethport, NJ, died on August 1 after a brief illness. He had been associated with the company for more than 40 years.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